SUMMARY REPORT ON THE CITY OF ST. LOUIS PARK ACTIVATED CARBON PILOT PLANT STUDY JULY AND OCTOBER, 1979

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Dated: January 11, 1980

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I. INTRODUCTION

The City of St. Louis Park conducted two small-scale pilot studies to determine the feasibility of using powdered activated carbon (PAC) for removing polynuclear aromatic compounds (PNA) (creosote contamination) from the drinking water supply. One test run was conducted in July, 1979 and the second test run was conducted in October, 1979.

Carbon slurry was pumped into the drinking water at the head of well No. 15. The well was operated at 1,000 gpm capacity. The drinking water-carbon mixture traveled several hundred feet to high pressure sand filters where the carbon was filtered from the water.

To test the effectiveness of PNA removal, water samples were collected after sand filtration and analyzed using high performance liquid chromatography (HPLC) techniques. Post filtration values were compared to non-PAC treated values.

what about October non-PAC values?

In the July test, two different PAC concentration levels were tried to determine the effect of carbon levels on PNA removal. They were approximately 2 mg/l and 10 mg/l. The Minnesota Department of Health (MDH) was responsible for the analysis of the water samples. SERCO Laboratories analyzed nine split samples.

In October, a second smaller study was conducted because the PAC slurry pump caused problems in the first test of July. The PAC concentration was set at 10.87 mg/l; and eight samples were collected and analyzed by SERCO Laboratories.

SERCO Laboratories provided analytical and technical assistance in these pilot studies.

This report summarizes the results of the two studies.

II. ANALYTICAL METHODOLOGY

The analysis for polynuclear aromatic hydrocarbons (PAH) can be done using either gas chromatography techniques (GC) or high performance liquid chromatography techniques (HPLC). SERCO Laboratories and the Minnesota Department of Health both use HPLC techniques.

SERCO Laboratories' HPLC instrument is a Hewlett-Packard (HPLC) model 1084B. It is equipped with an automatic sampling system, which can hold 60 samples, a programmable mirro-processor, two different detectors and a built-in digital integrator printer/plotter. The detectors are a HP79875A programmable variable wave length UV-visible scanner, and a Schoeffel spectrofluorometer, model FS-970, for detection of fluorescence compounds.

The sampling and analysis procedure employed by SERCO follows the proposed EPA test procedure, No. 610, as published in the December 3, 1979 issue of the Federal Register, Volume 44, No. 233.

PAH's are a class of ring-type compounds that can number, perhaps, into the hundreds. SERCO only analyzes for those as listed in EPA test

procedure 610, as stated above. At the present time, 12 of the 16 compounds listed are analyzed. MDH analyzes for 6 on that list but in addition analyzes for other PAH compounds (see enclosed MDH data sheets for list of compounds). A copy of the test procedure is enclosed for reference purposes.

III. RESULTS AND DISCUSSION OF THE JULY STUDY

The pilot studies of July and October were conducted by Mr. Vern Tollefsrud, City of St. Louis Park. The design of the July pilot study was a joint effort amongst SERCO Laboratories, Minnesota Department of Health, and the City of St. Louis Park. Samples were collected, by Mr. Tollefsrud, in amber-colored solvent bottles. MDH was primarily responsible for the analysis of the July samples. SERCO Laboratories analyzed nine split samples.

In the July study, temperature and pH were recorded at the time of sampling because these two factors are involved in sorption phenomena. Temperature and pH remained constant throughout the study (Table 1), at 11°C and 6.5 respectively. SERCO Laboratories also analyzed samples for suspended solids (Table 1) as a means to monitor the carbon slurry content. Due to slurry-pump problems, the first phase of the study had PAC concentrations that ranged between 1 and 4 mg/l; in the second phase, the concentrations ranged between 9 and 12 mg/l (two samples).

You never mention the significance of the data, & there anything significant?

Table 1 shows MDH results. Results are grouped as: Untreated (sampled at well head); Post-sand filter, untreated; Post-sand filter, treated (carbon slurry 1 to 4 mg/l - Phase I); Post-sand filter, treated (carbon slurry 9-12 mg/l). PAH parameters listed are only those which are also analyzed for by SERCO Laboratories.

Untreated samples show positive results for acenapthene, phenanthrene/pyrene, and fluoranthene. Chrysene shows negative. All values are given as nanograms per liter (ng/l); also can be stated as parts per trillion.

Untreated samples were collected after passage through the sand-filter to determine effect of filtration on PAH removal. It appears that fluoranthene, and perhaps, phenanthrene (pyrene co-elutes with phenanthrene and then it cannot be determined here which compound is present) are affected by filtration.

In what way?

The presence of PAC does effect PAH removal at the 1-4 mg/l level of PAC; phenanthrene is reduced from approximately 1500 ng/l to 200-300 ng/l. At the 9-12 mg/l, the level of PAH appears to be significantly reduced.

The values for anthracene, pyrene and fluoranthene obtained for well No. 15 by the MDH in May-August, 1978 are included (Table 1) for sake of comparison. These values generally confirm the July values and are more than likely representative of PAH levels in well No. 15.

SERCO's results for untreated samples (Table 2) generally confirm the presences of these compounds at the levels indicated. Untreated samples for May 29, 1979 (earlier exploratory analysis) and 11:00 A.M., July 16, 1979 agree quite closely to MDH results. There are some discrepancies which cannot be explained. Discrepancies can be due to either sampling and analysis techniques, or due to actual variations in the sample, or both. Sampling techniques themselves can introduce error. One potential source of error is the sorption of PAH compounds on the container wall. This error can be reduced if the entire container is extracted, rather than in the case where a sample is removed from the glass bottle and then extracted. Recently SERCO initiated the total container extraction step.

There are enormous discrepancies between MDH and SERCO.

Analytically, the PAH analysis is considered very difficult and complex. The very low level of analysis that is required and the nature of the methodology involved in themselves requires exacting techniques and extreme care. Also, day-to-day instrument vagaries, oftentimes beyond the control of the analyst, can create some uncertainties.

These caveates are offered not to create doubt, but to provide insight (and perhaps forbearance) while viewing PAH data.

MDH seems to be more useful in their data. See Table 1

SERCO's treated water results (Table 2) for the July pilot test generally show less-than values for all parameters. In comparison, the MDH results show positive values. Again, there is no ready explanation for the apparent difference, however, analytical problems are likely the cause.

What use are SERCOS results with the data as reported in Table 2?

Another important aspect to consider in viewing PAH data are the chromatograms. Chromatograms are graph-like results produced by the responses of the instrument to the PAH compounds when they elute after passage through the detector. Included in this report are the chromatograms resulting from a solution of PAH standards. PAH compounds in samples are identified if peaks in the samples elute at a time concurrent with a particular standard compound. If a compound elutes at a time that does not match a standard, this compound is not identified nor recorded.

Thus, it is important to realize that compounds other than those tabulated likely exist in the water. Some chromatograms are included to demonstrate this fact. How did you select the attachments? Significance of Chromatograms?

IV. RESULTS AND DISCUSSION OF THE OCTOBER STUDY

Where is the HHDH data for the October Study

Because slurry-pump problems were encountered in the July study, a second but reduced study was conducted in October. The PAC level was set for 10.87 mg/l concentration. Eight samples were collected by St. Louis Park, and analyzed by SERCO Laboratories.

Temperature, suspended solids and pH were not recorded in this study. Table 2 shows the results of that study. In the last two samples, well No. 11 was added to the system. Why? To see the effect on PAH + Carbon Treatment on a diluted water — 11-15 Dent

→ The numerical results are all less than detection. The attached chromatograms, however, do show the continued presence, albeit low, of some forms of PAH

this statement can be made about all of SERCO tests after 5/24/79

In that test only % of samples were less than detection.

In the remaining tests thru October 10/76 - all the samples show detectable results, treated or untreated.

compounds. Because the untreated water sample results do not compare with previous untreated results, the results for the treated samples are uncertain. Although examination of the chromatograms suggest that the treated water sample results show the effect of PAC on PAH removal.

How do you know which PAH compounds are removed and to what degree by viewing the chromatographs?

V. CONCLUSIONS

The purpose of the two small-scale pilot studies was to determine the feasibility of using powdered activated carbon (PAC) for removing polynuclear aromatic ompounds (PAH) from drinking water. Conversely, these studies should not be viewed as an attempt to develop all necessary information which would be required prior to the design and implementation of a full-scale treatment plant.

Based on the results presented herein, PAC will remove PNA compounds from water. Further, it appears that the basic design of PAC introduction into the system, and the subsequent removal of PAC by sand-filter, is adequate. However, a detailed study of the system is warranted to insure maximum efficiency in removal and to keep costs to a minimum. Sorption of organic compounds onto activated carbon is a complex, not always a well understood, reaction which involves many factors. Because of differences in molecular characteristics, not all PAH compounds would be expected to sorb at the same level.

Very conservative statement - is this a result of the limited testing?

Thus, a more detailed pilot study is warranted in order to develop more specific information. Basically, the sought after information is "What is

the minimum amount of PAC material required to remove specified PAH compounds down to a specified level?" and "What will the treatment costs be?"

Submitted by:

SERCO LABORATORIES

Daryle A. Thingvold, Ph.D.
Technical Director

Based on the July & October pilot treatment studies SERCO is not equipped to develop the more specific inform. needed to answer these two questions: Is anyone?

TABLE 1

ST. LOUIS PARK
PILOT CARBON TREATMENT STUDY
JULY 16-20, 1979
WELL NUMBER 15
MINNESOTA DEPARTMENT OF HEALTH's RESULTS
SERCO Laboratories January 9, 1980

Test Condition	Partial List of Parameters (ng/l)					
	Acenapthene	Anthracene	Phenanthrene/Pyrene*	Fluorene	Fluoranthene	Chrysene
Untreated Samples						
7/16 11:00 AM	3200	-	1700	-	510	<25
7/18 11:00 AM	2400	-	1800	-	440	<25
7/19 5:00 AM	2800	-	1900	-	380	<25
7/20 5:00 AM	<2.2	60	1400	-	190	-
Post Sand-Filter Untreated						
7/16 11:05 AM	<140	-	6.1	-	3.6	<25
7/16 11:00 PM	2500	-	1900	-	84	<25
7/16 5:00 PM	1600	-	440	-	91	<25
7/19 3:00 AM	4800	-	1100	-	32	<25
7/19 3:00 AM (Dupl.)	3900	-	1000	-	32	<25

* co-eluting compounds
< means "less than"

TABLE 1

ST. LOUIS PARK
PILOT CARBON TREATMENT STUDY
JULY 16-20, 1979
WELL NUMBER 15
MINNESOTA DEPARTMENT OF HEALTH's RESULTS
SERCO Laboratories January 9, 1980

Test Condition	Partial List of Parameters (ng/l)					
	Acenapthene	Anthracene	Phenanthrene/Pyrene*	Fluorene	Fluoranthene	Chrysene
Post Sand-Filter Treated, 1-2 mg/l						
7/17 11:00 AM	910	-	430	-	89	<25
7/17 11:00 AM (Dupl.)	1200	-	360	-	81	<25
7/17 5:00 PM	1300	-	300	-	62	<25
7/17 11:00 PM	170	<8.0	180	-	39	<25
7/17 11:00 PM (Dupl.)	2700	-	190	-	30	<25
7/18 5:00 AM	1500	-	180	-	28	<25
7/18 11:00 AM	2600	-	210	-	41	<25
7/18 5:00 PM	190	<8.0	210	-	17	-
7/18 9:00 PM	190	<8.0	240	-	15	-

* co-eluting compounds
< means "less than"

TABLE 1

ST. LOUIS PARK
PILOT CARBON TREATMENT STUDY
JULY 16-20, 1979
WELL NUMBER 15
MINNESOTA DEPARTMENT OF HEALTH's RESULTS
SERCO Laboratories January 9, 1980

Test Condition	Partial List of Parameters (ng/l)					
	Acenapthene	Anthracene	Phenanthrene/Pyrene*	Fluorene	Fluoranthene	Chrysene
Post-Sand Filter Treated, 9-12 mg/l						
7/19 5:00 AM	110	<8.0	510	-	14	<25
7/19 11:00 AM	21	<8.0	10	-	7.0	-
7/19 5:00 PM	<2.2	<8.0	27	-	3.6	-
7/19 11:00 PM	<2.2	<8.0	<1.0	-	1.0	-
7/20 5:00 AM	<2.2	<8.0	<1.0	-	1.2	-
May-August 1978	-	190	750[1]	-	390	-
	-	241	1221[1]	-	292	-

* co-eluting compounds

[1] Pyrene only

< means "less than"

```
 55.00   %B          40.0
 60.00   STOP
%B    6 0 @
%B   *6 0 ESCAPE
TIME 5 %B    6 0 @
TIME 3 0 %B    5 CLEAR #
3 5 @
TIME 4 0 %B    8 5 @
TIME 5 0 %B    6 0 @
TIME 5 5 STOP @
INJ      START  MESSAGE  8
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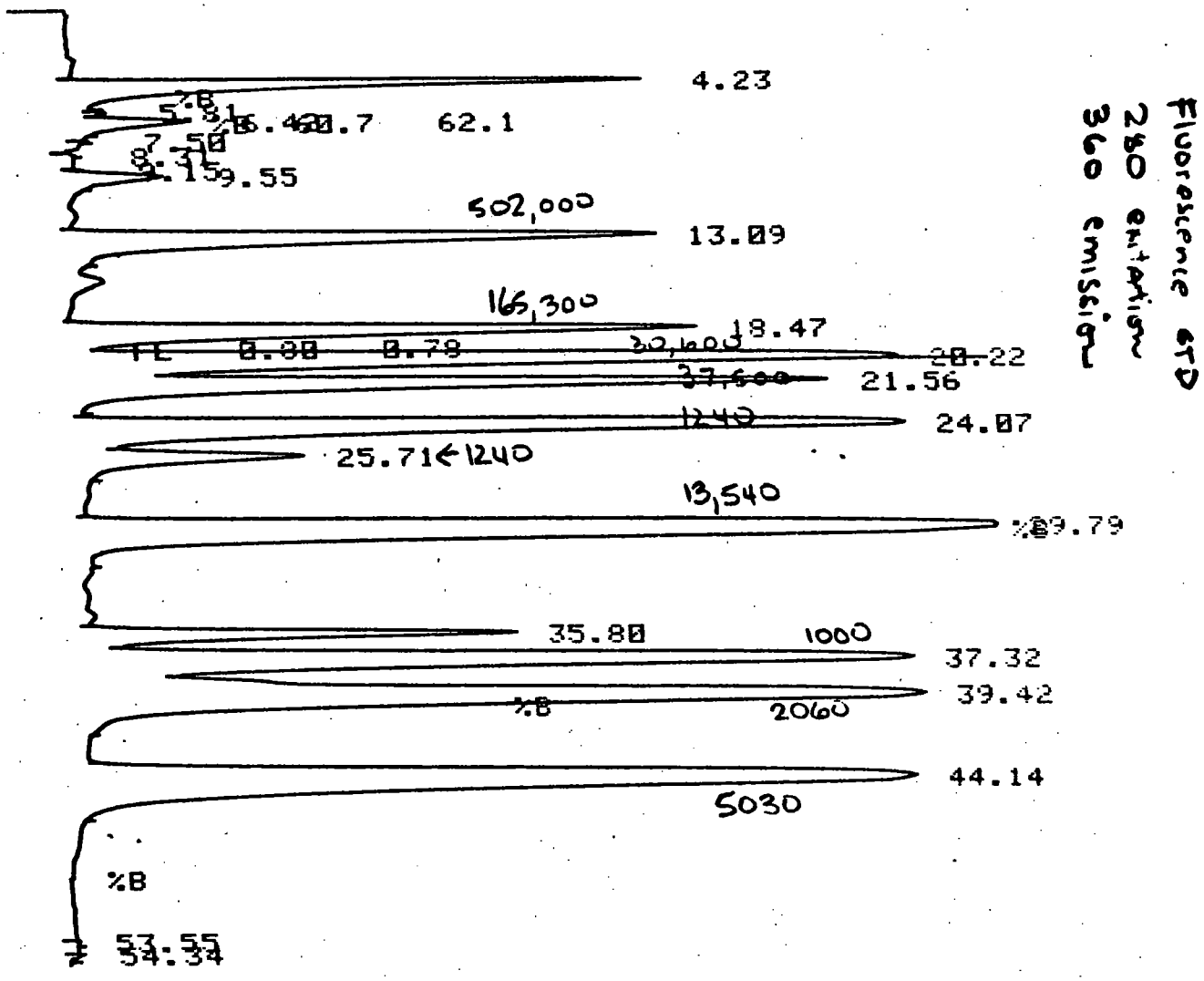


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ID:80
NO CALIB
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6.42	79040	59.401
9.55	80600	60.574
13.09	729000	547.867
18.47	769800	578.530
20.22	1223000	919.125
21.56	933800	701.781
24.07	1246000	936.410
25.71	256600	192.843
29.79	1845000	1386.58
35.80	411400	309.181
37.32	1408000	1058.16
39.42	1793000	1347.50
44.14	1999000	1502.31

ADY
INJ

START SCAN

FL 1.00 0.98

%B 280 : 430

28.529

14.37

19.61

ATTN92↑ 7 9

23.89

%B

37.91

%B
40.76

STOP44

UV-STD
280nm

APPENDIX B

LABORATORY REPORTS AND CHROMATOGRAMS FOR JULY STUDY

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2982 N. Cleveland Ave. Roseville, Mn. 55113 (612) 636-7173

REPORT OF LABORATORY ANALYSIS
(Methodologies EPA approved)

REPORT NO: 1480 PAGE 2 OF 2
08/20/79

LIENT: City of St. Louis Park
ATE COLLECTED: 07/16/79 COLLECTED BY: CLIENT
ATE RECEIVED: 07/16/79 PICKED-UP BY: SERCO
AMPLE DESCRIPTION: WELL WATER

	LAB NO: 4979
	SAMPLE SITE: WELL WATER
NALYSIS:	
uspended Solids, mg/l	2
H	6.5
emperature, °C	11
Acenaphthene, ng/l	**
Anthracene, ng/l	<9.3
Benzo(a)anthracene, ng/l	<0.2
Benzo(a)pyrene, ng/l	510
Benzo(ghi)perylene, ng/l	<0.6
Chrysene, ng/l	<0.2
Dibenzo(ah)anthracene, ng/l	<0.3
Fluorene, ng/l	**
Fluoranthene, ng/l	340
Naphthalene, ng/l	<170
Phenanthrene, ng/l	<7.0
Pyrene, ng/l	460

** These peaks co-elute -- the total result is 17 ng/l.

roved b : < means "less than"

2.13		9746	1.045
2.96		624300	6.694
4.13		126500	1.356
4.68		119800	1.285
15.76	.0016	3136	0.034

DF: 1.6000 E- 3

F1

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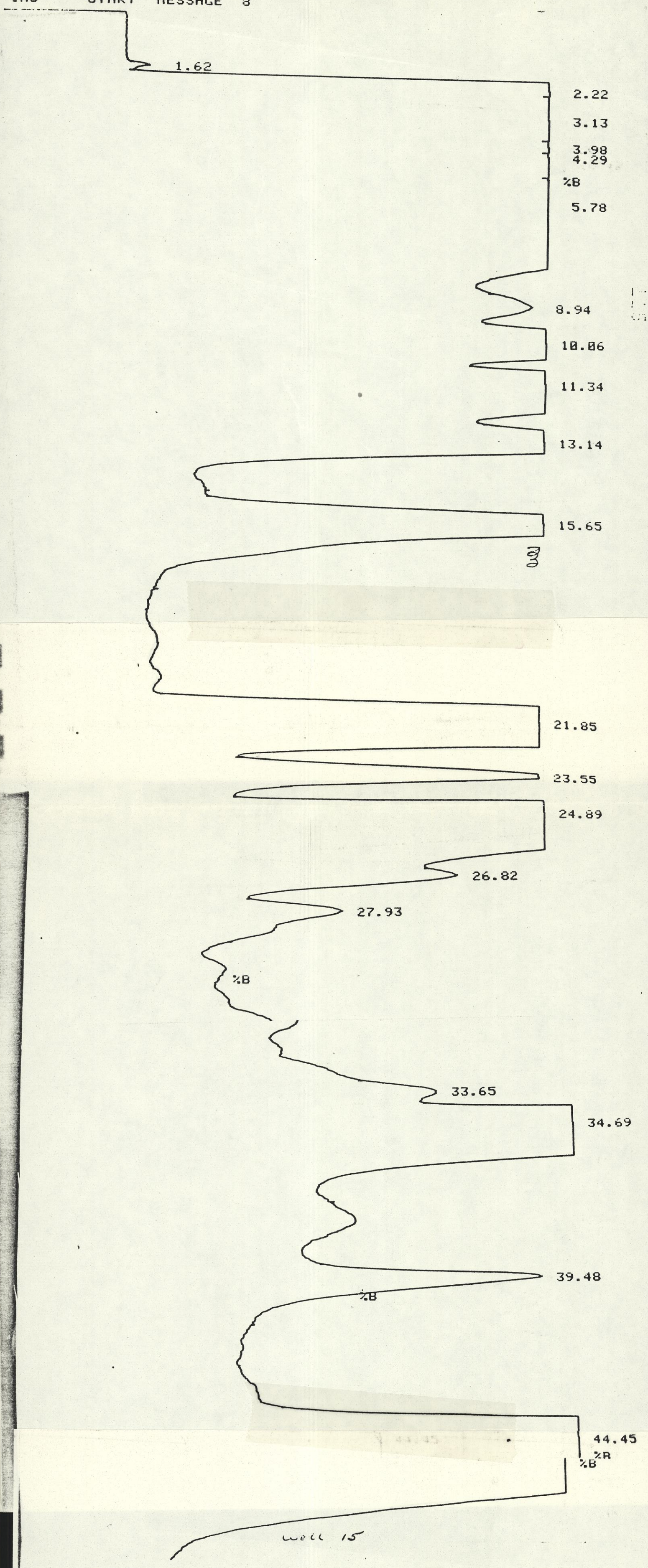


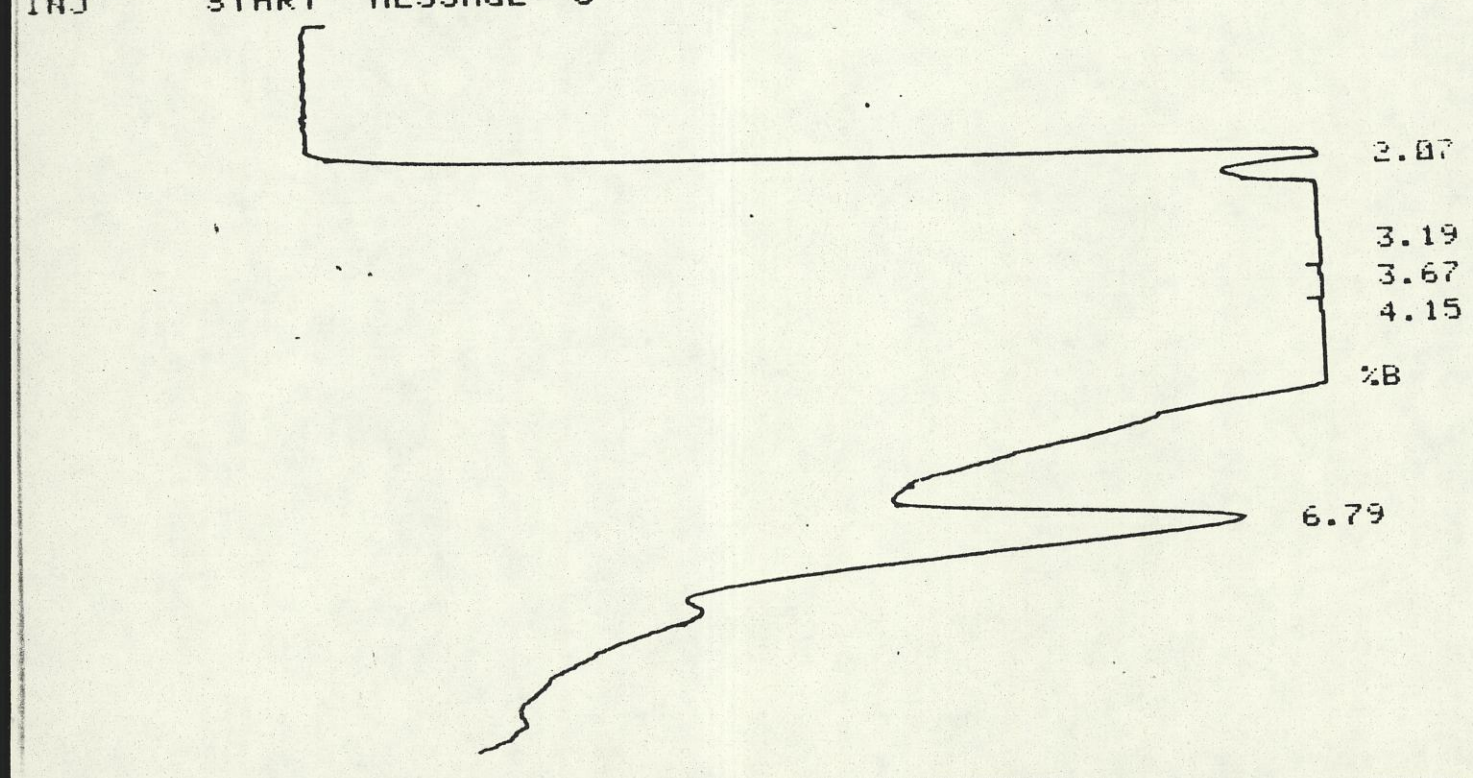
hp 1080 B

BIL: 29
ID:4979 Well Water
ESTD FILE 2

RT	EXP RT	AREA	CAL #	AMT
2.22		156200		1.099
3.13		1139000		8.015
3.98		230800		1.624
4.29		362300		2.549
5.78		1331000		9.366
8.94		191800		1.350
10.06		321600		2.263
11.34	11.27	510900	2	102.961
13.14		253600		1.785
15.65	~16.33	273300		1.923 17
21.85	21.90	1222000	5	0.340
23.55	23.59	121400	6	0.180
24.89		1142000		8.036
26.82		79000		0.556
27.93	28.10	45790	7	0.076
33.65	33.52	19300	8	0.037
34.69		1494000		10.513
39.48		70260		0.494
44.45		848800		5.973

DF: 1.0500 E- 3

INJ START MESSAGE 8



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REPORT OF LABORATORY ANALYSIS
(Methodologies EPA approved)

ENT: City of St. Louis Park
E COLLECTED: 07/17/79 COLLECTED BY: CLIENT
E RECEIVED: 07/19/79 PICKED-UP BY: CLIENT
PLE DESCRIPTION: WASTEWATER

	LAB NO:	5037
	SAMPLE SITE:	WELL#15 TREATED 11:00AM
LYSIS:		7/17/79
naphthene, ng/l		*
thracene, ng/l		<10
nzo(a)anthracene, ng/l		<0.2
nzo(a)pyrene, ng/l		<0.2
nzo(ghi)perylene, ng/l		<0.7
rysene, ng/l		<0.2
benzo(ah)anthracene, ng/l		<0.4
uorene, ng/l		*
uoranthene, ng/l		<0.3
phtnalene, ng/l		<180
enanthrene, ng/l		<7.7
rene, ng/l		<1.6

* These peaks co-elute -- the total result is 54 ng/l.

proved by: [signature] < means "less than"

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REPORT OF LABORATORY ANALYSIS
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REPORT NO: 1501
08/17/79

LIENT: City of St. Louis Park
ATE COLLECTED: 07/17-18/79
ATE RECEIVED: 07/18/79
AMPLE DESCRIPTION: WELL WATER

COLLECTED BY: CLIENT
PICKED-UP BY: CLIENT

LAB NO:	5017	5018
SAMPLE SITE:	WELL #15 5:00 PM	WELL #15 5:00 PM
NALYSIS:	7/17/79	7/18/79
cenaphthene, ng/l	<44	<79
nthracene, ng/l	<6.6	<12
enzo(a)anthracene, ng/l	<1.2	<2.2
enzo(a)pyrene, ng/l	<0.1	<0.2
enzo(ghi)perylene, ng/l	<0.2	<0.4
nrysene, ng/l	<1.2	<2.2
ibenzo(ah)anthracene, ng/l	<0.2	<0.4
luorene, ng/l	<44	<79
luoranthene, ng/l	<0.2	<0.4
aphthalene, ng/l	<120	<210
henanthrene, ng/l	<5.0	<9.0
yrene, ng/l	<1.1	<2.0

pproved by:

< means "less than"

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REPORT OF LABORATORY ANALYSIS
(Methodologies EPA approved)

REPORT NO: 1524
08/20/79

ENT: City of St. Louis Park
E COLLECTED: 07/19/79
E RECEIVED: 07/19/79
PLE DESCRIPTION: WASTEWATER

COLLECTED BY: CLIENT
PICKED-UP BY: SERCO

	LAB NO:	5067
	SAMPLE SITE:	WELL #15 5:00 AM
LYSIS:		7/19/79
naphthene, ng/l		<49
hracene, ng/l		<7.5
zo(a)anthracene, ng/l		<0.1
zo(a)pyrene, ng/l		<0.1
zo(ghi)perylene, ng/l		<0.5
ysene, ng/l		<0.1
enzo(ah)anthracene, ng/l		<0.3
ɔrene, ng/l		<49
ɔranthene, ng/l		0.4
hthalene, ng/l		135
nanthrene, ng/l		<5.7
ene, ng/l		<1.2

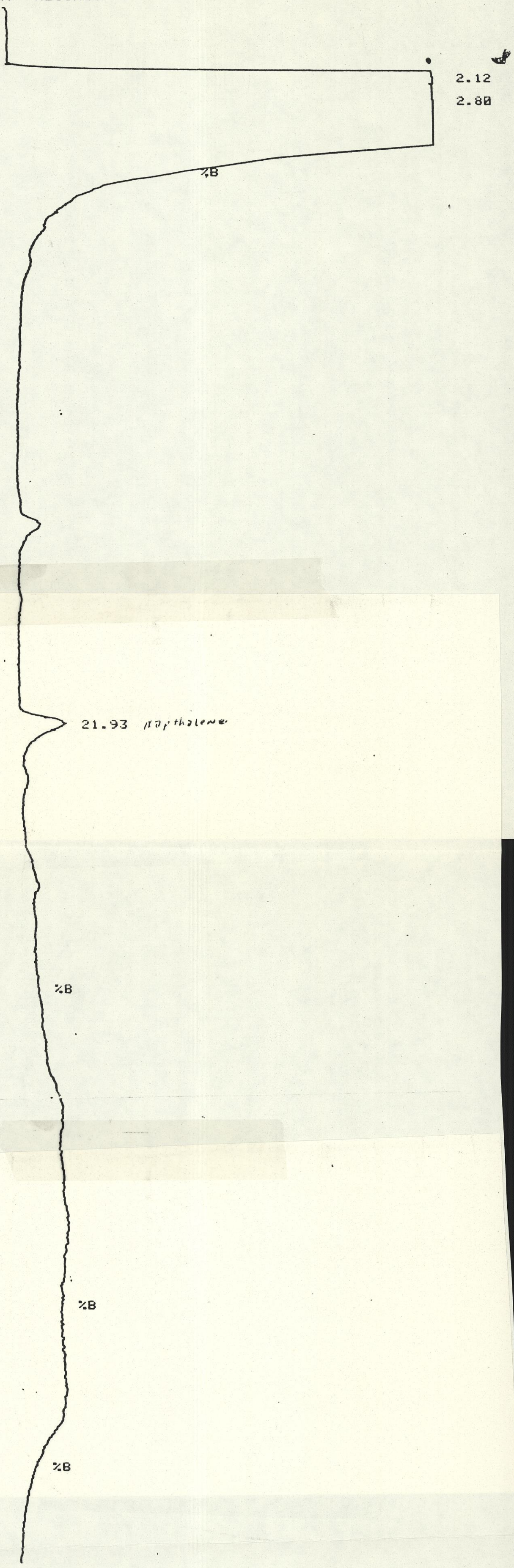
ved by: DR

< means "less than"

33.53 33.52

DF: 1.0000 E- 3

INJ START MESSAGE 8



hp 1080 B

BTL: 32
ID:5067 Well 15
ESTD FILE 2

RT	EXP RT	AREA	CAL #	AMT
2.12		211000		1.202
2.80		1063000		6.055
21.93	21.90	1618	5	0.000 3643

DF: 8.5000 E- 4

INJ START MESSAGE 8

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REPORT OF LABORATORY ANALYSIS
(Methodologies EPA approved)

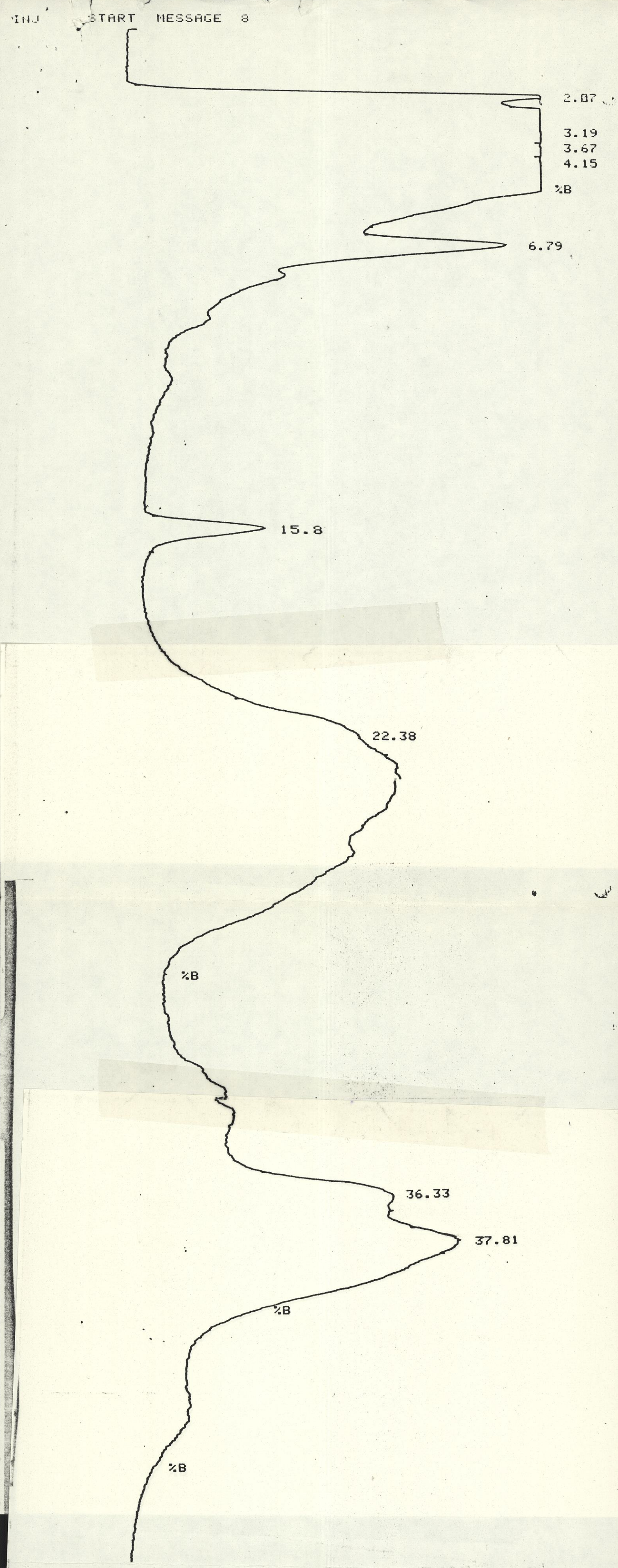
REPORT NO: 1534
08/20/79

CLIENT: City of St. Louis Park
DATE COLLECTED: 07/20/79
DATE RECEIVED: 07/20/79
SAMPLE DESCRIPTION: WASTEWATER

COLLECTED BY: CLIENT
PICKED-UP BY: CLIENT

ANALYSIS:	LAB NO: 5095 SAMPLE SITE: WELL#15 RAW 5:00 AM 7/20/79	5096 WELL#15 TREATED 5:00 AM 7/20/79
Acenaphthene, ng/l	<87	<58
Anthracene, ng/l	<9.3	<8.9
Benzo(a)anthracene, ng/l	<0.7	<0.2
Benzo(a)pyrene, ng/l	4.0	<0.2
Benzo(ghi)perylene, ng/l	<0.9	<0.6
Chrysene, ng/l	<0.7	<0.2
Dibenzo(ah)anthracene, ng/l	4.0	<0.3
Fluorene, ng/l	<87	<58
Fluoranthene, ng/l	2.0	<0.3
Naphthalene, ng/l	<240	<160
Phenanthrene, ng/l	<10	<6.7
Pyrene, ng/l	<1.5	<1.4

Approved by: _____ < means "less than"



hp 1080 B

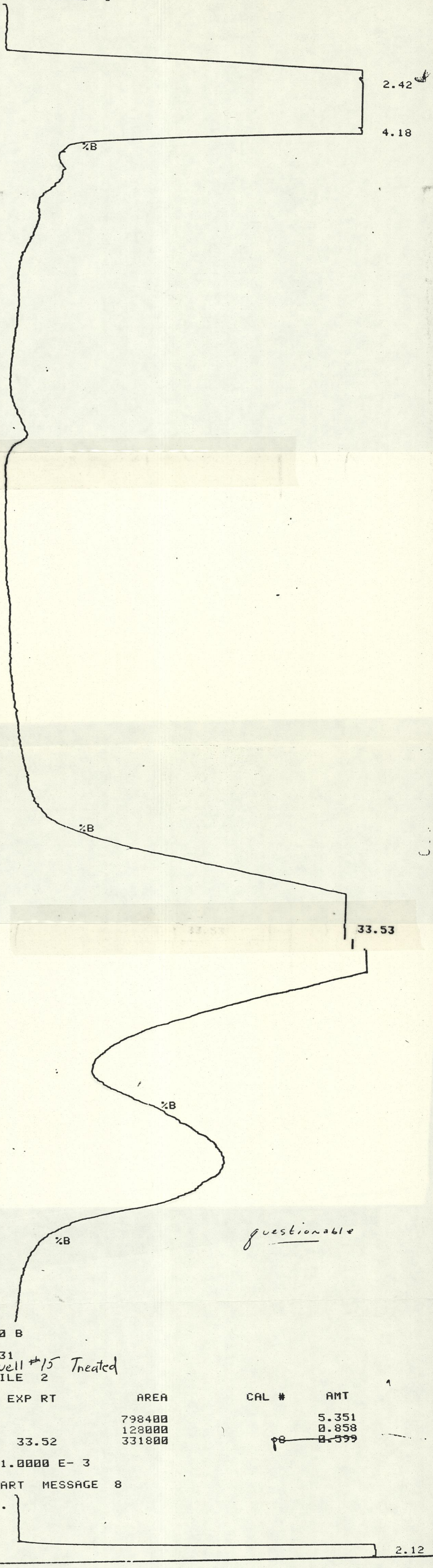
BTL: 30
~~11:5095~~ Well #15 Raw
ESTD FILE 2

RT	EXP RT	AREA	CAL #	AMT
2.07		68700		0.691
3.19		343400		3.452
3.67		126400		1.271
4.15		285700		2.872
6.79		39920		0.401
15.82		27110		0.273
22.38	21.90	4237	5	0.002
36.33	35.97	16170	9	0.004
37.81	38.20	8430	10	0.004

DF: 1.5000 E- 3

INJ START MESSAGE 8

INJ START MESSAGE 8



guestionable

hp 1080 B

BTL: 31
ID:5096 well #15 Treated
ESTD FILE 2

RT	EXP RT	AREA	CAL #	AMT
2.42		798400		5.351
4.18		128000		0.858
33.53	33.52	331800	~~8~~	~~0.599~~

DF: 1.0000 E- 3

INJ START MESSAGE 8

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REPORT OF LABORATORY ANALYSIS
(Methodologies EPA approved)

REPORT NO: 1533
07/31/79

IENT: City of St. Louis Park
TE COLLECTED: 07/19-29/79
TE RECEIVED: 07/20/79
MPLE DESCRIPTION: WASTEWATER

COLLECTED BY: CLIENT
PICKED-UP BY: CLIENT

LAB NO:	5091	5092	5093	5094
SAMPLE SITE:	WELL #15 11:00AM 7/19/79	WELL #15 5:00 PM 7/19/79	WELL #15 11:00PM 7/19/79	WELL #15 5:00 AM 7/20/79
ALYSIS:				
spended Solids, mg/l	12	11	13	12
mperature, °C	10	10	10	10
	7.4	7.3	6.0	6.5

proved by: DE — < means "less than"

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REPORT OF LABORATORY ANALYSIS
(Methodologies EPA approved)

REPORT NO: 1509
07/31/79

CLIENT: City of St. Louis Park
DATE COLLECTED: 07/18-19/79
DATE RECEIVED: 07/19/79
SAMPLE DESCRIPTION: WASTEWATER

COLLECTED BY: CLIENT
PICKED-UP BY: CLIENT

LAB NO:	5034	5035	5036
SAMPLE SITE:	WELL #15 5:00 PM 7/18/79	WELL #15 9:00 PM 7/18/79	WELL #15 5:00 AM 7/19/79
ANALYSIS:			
Suspended Solids, mg/l	4	1	9
pH	6.5	6.5	6.5
emperature, °C	10	10	10

pproved b :

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REPORT OF LABORATORY ANALYSIS
(Methodologies EPA approved)

REPORT NO: 1502
07/25/79

T: City of St. Louis Park
COLLECTED: 07/16-18/79
RECEIVED: 07/18/79
LE DESCRIPTION: WELL WATER

COLLECTED BY: CLIENT
PICKED-UP BY: CLIENT

LAB NO:	5019	5020	5021	5022	5023
SAMPLE SITE:	11:00PM 7/16/79	5:00 AM 7/17/79	11:00AM 7/17/79	5:00 PM 7/17/79	11:00PM 7/17/79
LYSIS:					
pended Solids, mg/l	1	2	1	1	1
	6.5	6.6	6.6	6.5	6.5
perature, °C	10	10	10	10	10

LAB NO:	5024
SAMPLE SITE:	5:00 AM 7/18/79
LYSIS:	
pended Solids, mg/l	1
	6.5
perature, °C	10

proved by: _____ < means "less than"

APPENDIX C

LABORATORY REPORTS AND CHROMATOGRAMS FOR OCTOBER STUDY

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REPORT OF LABORATORY ANALYSIS
(Methodologies EPA approved)

REPORT NO: 2119
10/22/79

IENT: City of St. Louis Park
TE COLLECTED: 10/02/79 COLLECTED BY: CLIENT
TE RECEIVED: 10/02/79 PICKED-UP BY: CLIENT
MPLE DESCRIPTION: WELL WATER

LAB NO:	6857	6858
SAMPLE SITE:	WELL 15 8:00 AM RAW	WELL 15 2:00 PM TREATED
NALYSIS:	10/2/79	10/2/79
cenaphthene, ng/l	<390 **	<470 **
thracene, ng/l	<190	<220
enzo(a)anthracene, ng/l	<13 *	<16 *
nzo(a)pyrene, ng/l	<4	<4
enzo(ghi)perylene, ng/l	<13	<16
hrysene, ng/l	<13 *	<16 *
ibenzo(ah)anthracene, ng/l	<6	<8
luorene, ng/l	<390 **	<470 **
luoranthene, ng/l	<5	<6
aphthalene, ng/l	<320	<380
henanthrene, ng/l	<110	<140
yrene, ng/l	<21	<24

* The peaks for benzo-a-anthracene and chrysene co-elute -- the result given is the total result for these two compounds.

* The peaks for acenaphthene and fluorene co-elute -- the result given is the total result for these two compounds.

pproved by: < means "less than"

START MESSAGE 8

2.03

4.34

%B

5.36

9.21

12.00

14.59

17.45

19.90

21.35

23.28
23.97

%B

%B

%B

6857 4.2ml/t 100ul FL DET

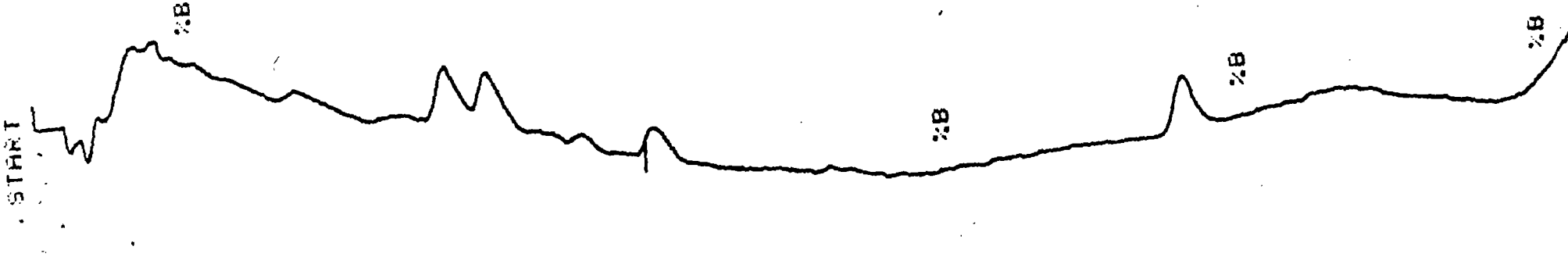
PRETREATMENT

10/2/14

10/2/79

6857 4.2mg/L 100ul inj UV DET.

PRE TREATMENT



EXT SGNL @
EXT SGNL
STOPN 28 6 @
INJ START

%B

%B

6858 Smpl#2 1000 ng FL det.

Post Treatment

10/2/19

SAMPLE 6858 100ml inj

Conc 5mg/ml

10/2/79

POST TREATMENT UV DET

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REPORT OF LABORATORY ANALYSIS
(Methodologies EPA approved)

REPORT NO: 2147
10/22/79

#1568/

ENT: City of St. Louis Park
E COLLECTED: 10/03/79
E RECEIVED: 10/03/79
MPLE DESCRIPTION: WELL WATER

COLLECTED BY: CLIENT
PICKED-UP BY: CLIENT

	Peru	
LAB NO:	6930	6931
SAMPLE SITE:	WELL 15 2:00 AM TREATED	WELL 15 2:00 PM TREATED
LYSIS:	10/3/79	10/3/79
enaphthene, ng/l	<720 **	<420 **
hracene, ng/l	<340	<200
zo(a)anthracene, ng/l	<10 *	<15 *
nzo(a)pyrone, ng/l	<6	<4
nzo(ghi)perylene, ng/l	<10	<16
ysene, ng/l	<10 *	<15 *
benzo(ah)anthracene, ng/l	<2	<7
rene, ng/l	<720 **	<420 **
oranthene, ng/l	<9	<5
phthalene, ng/l	<590	<340
nanthrene, ng/l	<210	<120
rene, ng/l	<37	<21

The peaks for benzo-a-anthracene and chrysene co-elute -- the result given is the total result for these two compounds.

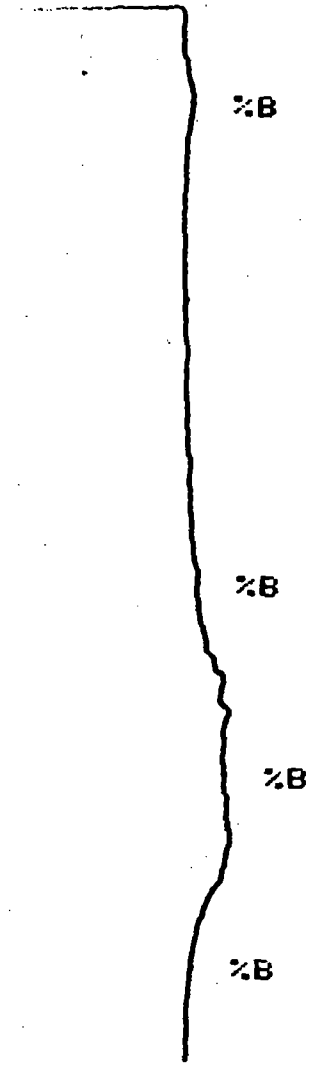
The peaks for acenaphthene and fluorene co-elute -- the result given is the total result for these two compounds.

roved by:

< means "less than"

DF: 1.0000 E+ 0

START MESSAGE 8

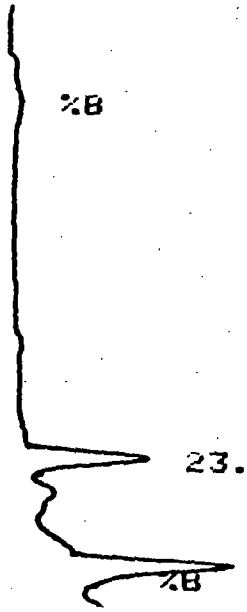


WELL #15 TREATED
2:00 PM. 10/3/79

P 1080 B

BTL: 2
-14-796930
?

START MESSAGE 8

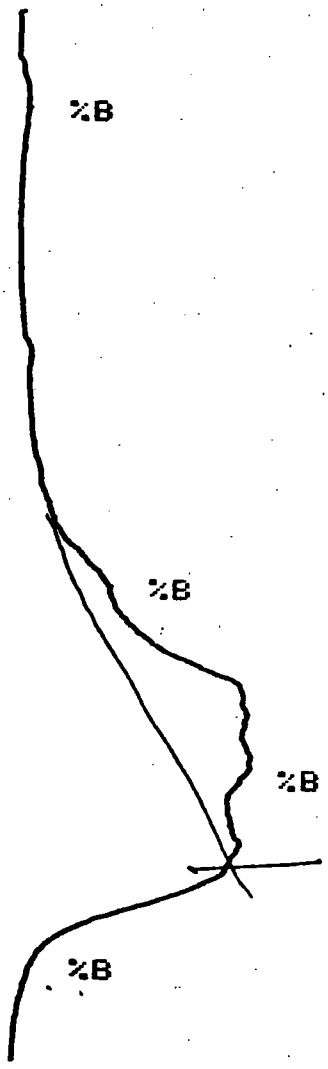


WELL #15+11 TREATED
2:00 PM. 10/4/79

23.66	23.70	60330	R	1	203.325
29.35	29.39	83600		7	916.426
36.75	36.75	52870		8	182.658
38.60	38.86	841		9	2.484

DF: 2.9000 E+ 0

INJ START MESSAGE 8



WELL #15 TREATED
2:00 A.M. 10/3/79

hp 1080 B

BTL: 4
ID:11-14-796931
AREAS?

INJ START MESSAGE 8

%B

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REPORT OF LABORATORY ANALYSIS
(Methodologies EPA approved)

REPORT NO: 2174
10/22/79

15483

LIENT: City of St. Louis Park
ATE COLLECTED: 10/04/79
ATE RECEIVED: 10/04/79
AMPLE DESCRIPTION: WELL WATER

COLLECTED BY: CLIENT
PICKED-UP BY: CLIENT

LAB NO:	7007	7008	7009	7010
SAMPLE SITE:	WELL#15 2:00 AM TREATED	WELL#15 12:00PM TREATED	11&15 2:00 PM TREATED	11&15 2:30 PM TREATED
NALYSIS:	10/4/79	10/4/79	10/4/79	10/4/79
Acenaphthene, ng/l	<360 **	<340 **	<430 **	<490 **
nthracene, ng/l	<170	<160	<200	<230
enzo(a)anthracene, ng/l	<13 *	<12 *	<15 *	<17 *
nzo(a)pyrene, ng/l	<4	<3	~~<4~~ 83	<5
enzo(ghi)perylene, ng/l	<12	<12	<15	<17
hrysene, ng/l	<13 *	<12 *	<15 *	<17 *
ibenzo(ah)anthracene, ng/l	<5	<3	<7	<8
luorene, ng/l	<360 **	<340 **	<430 **	<490 **
luoranthene, ng/l	<5	<5	~~46~~ 203	<7
Naphthalene, ng/l	<300	<280	<350	<400
henanthrene, ng/l	<110	<100	<120	<140
yrene, ng/l	<19	<18	<22	<26

* The peaks for benzo-a-anthracene and chrysene co-elute -- the result given is the total result for these two compounds.

** The peaks for acenaphthene and fluorene co-elute -- the result given is the total result for these two compounds.

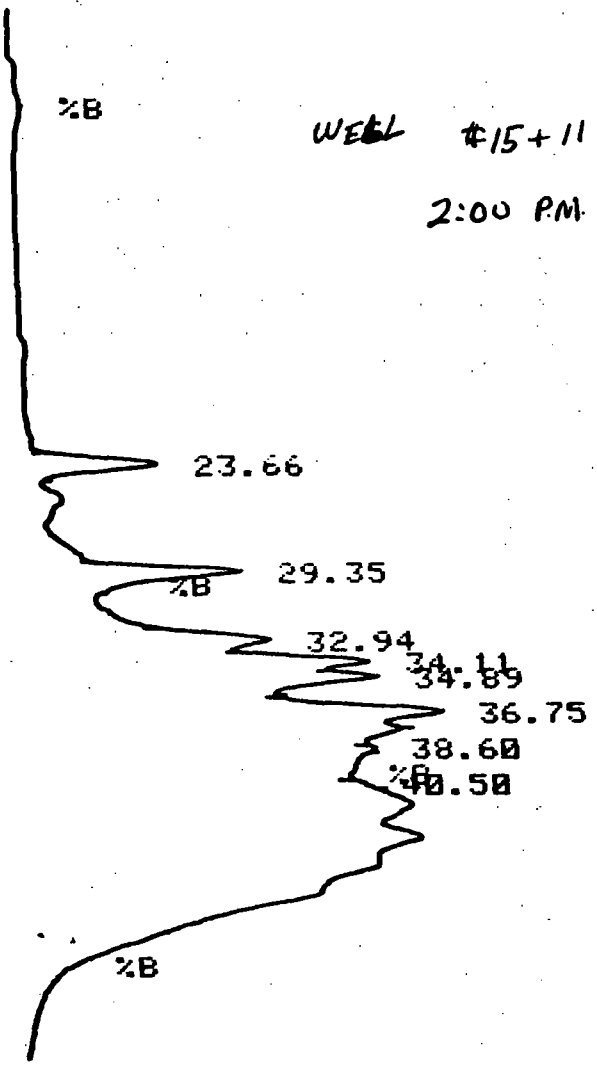
pproved by:

< means "less than"

hp 1080 B

BTL: 2
ID:11-14-796930
AREAS?

INJ START MESSAGE 8



WELL #15+11 TREATED
2:00 P.M. 10/4/79

hp 1080 B

BTL: 3
ID:11-14-797009
ESTD FILE 1

RT	EXP RT	AREA		CAL #	AMT
23.66	23.70	60330	R	1	203.325
29.35	29.39	83600		7	916.426
36.75	36.75	52870		8	182.658
38.60	38.86	841		9	2.484

DF: 2.9000 E+ 0

INJ START MESSAGE 8

WELL #15 TREATED
2:00 A.M. 10/3/79

%B

hp 1080 B

BTL: 8
ID:11-14-79A.01
AREAS?

INJ START MESSAGE 8

%B

Well #15
12:00 P.M.
Treated
10.87 ppm carbon

%B

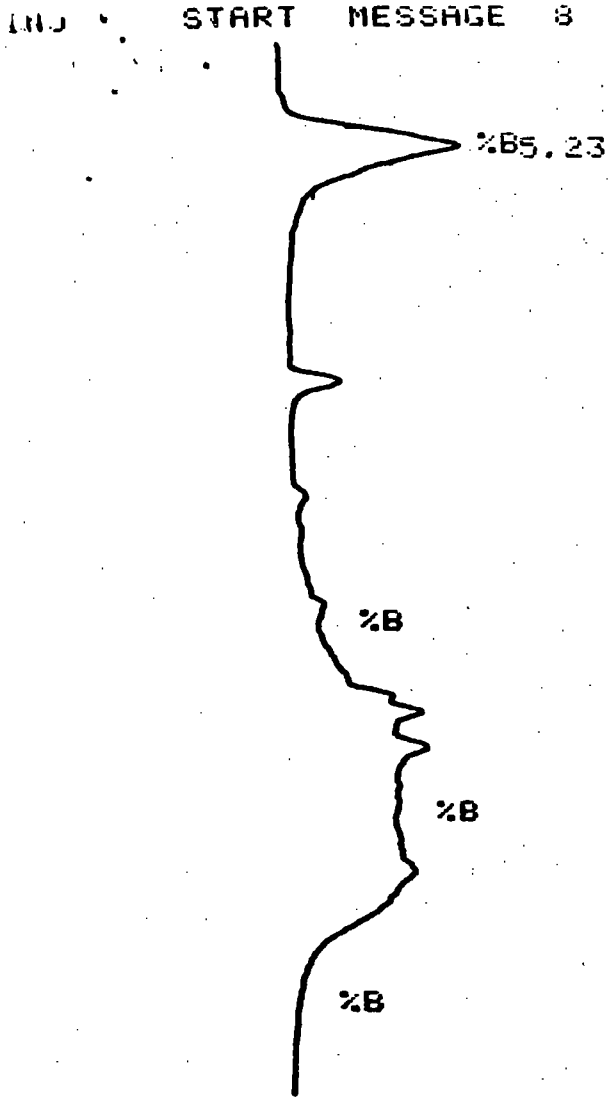
%B

%B

hp 1080 B

BTL: 9
ID:11-14-79A007
AREAS?

TOP
TOP @



11+15

2:30 TREATED

11-4-79

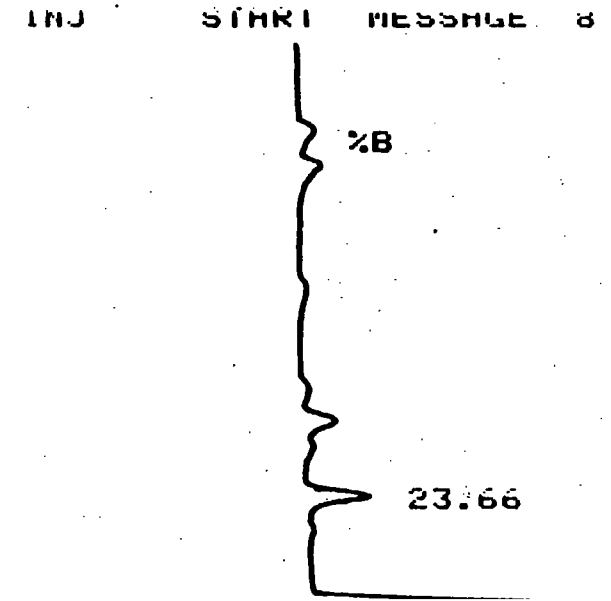
1081 ppm carbon

1080 B

BTL: 6
ID:11-14-79/7010.
NO PEAKS IN WDOS

RT	AREA	AREA %
5.23	70660	140.000

DF: 1.4000 E+ 0



APPENDIX D

RESULTS FOR WELL NUMBER 15

MAY 25, 1979 and NOVEMBER 11, 1979

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REPORT OF LABORATORY ANALYSIS
(Methodologies EPA approved)

REPORT NO: 2398 PAGE 2 OF 2
01/04/80

CLIENT: City of St. Louis Park
DATE COLLECTED: 11/01/79 COLLECTED BY: CLIENT
DATE RECEIVED: 11/01/79 PICKED-UP BY: CLIENT
SAMPLE DESCRIPTION: WELL WATER

	LAB NO:	7667
	SAMPLE SITE:	WELL#15 UNTREAT 9:30 AM
ANALYSIS:		11/1/79
Acenaphthene, ng/l		<50 **
Anthracene, ng/l		<50
Benzo(a)anthracene, ng/l		30 *
Benzo(a)pyrene, ng/l		<2
Benzo(ghi)perylene, ng/l		<40
Chrysene, ng/l		30 *
Dibenzo(ah)anthracene, ng/l		<5
Fluorene, ng/l		<50 **
Fluoranthene, ng/l		860
Naphthalene, ng/l		<100
Phenanthrene, ng/l		210
Pyrene, ng/l		400

* The peaks for benzo-a-anthracene and chrysene co-elute -- the result given is the total result for these two compounds.

** The peaks for acenaphthene and fluorene co-elute -- the result given is the total result for these two compounds.

Approved by: < means "less than"

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REPORT OF LABORATORY ANALYSIS
(Methodologies EPA approved)

REPORT NO: 1082 PAGE 2 OF 2
07/09/79

CLIENT: City of St. Louis Park
DATE COLLECTED: COLLECTED BY: CLIENT
DATE RECEIVED: 05/25/79 PICKED-UP BY:
SAMPLE DESCRIPTION: WELL WATER

	LAB NO: 3515
	SAMPLE SITE: WELL #15
ANALYSIS:	
Acenaphthene, ng/l	390
Acenaphthylene, ng/l	***
Anthracene, ng/l	170
Benzo(a)anthracene, ng/l	63
Benzo(a)pyrene, ng/l	1
Benzo(b)fluoranthene, ng/l	***
Benzo(ghi)perylene, ng/l	6.3
Benzo(k)fluoranthene, ng/l	***
Chrysene, ng/l	63
Dibenzo(ah)anthracene, ng/l	<0.57
Fluorene, ng/l	390
Fluoranthene, ng/l	290
Indeno(1,2,3-c,d)-pyrene, ng/l	***
Naphthalene, ng/l	<56
Phenanthrene, ng/l	160
Pyrene, ng/l	150

*** Analysis was not done.

Approved by: DR < means "less than"

APPENDIX E

RESULTS AND CHROMATOGRAMS FOR WELL NOS. 7, 9, 10 and 15

NOVEMBER 5, 1979

(RESULTS NOT CONFIRMED BY UV-DETECTOR)

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REPORT OF LABORATORY ANALYSIS
(Methodologies EPA approved)

CLIENT: City of St. Louis Park
DATE COLLECTED: 11/05/79 COLLECTED BY: CLIENT
DATE RECEIVED: 11/05/79 PICKED-UP BY: CLIENT
SAMPLE DESCRIPTION: LANDFILL MON. WELL

LAB NO:	7726	7727	7728	7729
SAMPLE SITE:	WELL #7 RAW WATER	WELL #9 RAW WATER	WELL#10 RAW WATER	WELL#15 RAW WATER
ANALYSIS:	11/5/79	11/5/79	11/5/79	11/5/79
Acenaphthene, ng/l	<150 **	<180 **	<140 **	<170 **
Benzo(a)pyrene, ng/l	<2	440	39	3.4
Chrysene, ng/l	<6 *	1400 *	2600 *	880 *
Anthracene, ng/l	<71	<84	<67	2400
Benzo(a)anthracene, ng/l	<6 *	1400 *	2600 *	880 *
Benzo(ghi)perylene, ng/l	<5	160	<5	<6
Dibenzo(ah)anthracene, ng/l	<3	88	<3	<3
Fluorene, ng/l	<150 **	<180 **	<140 **	<170 **
Fluoranthene, ng/l	5.9	400	3300	3200
Naphthalene, ng/l	<120	<150	<110	<140
Phenanthrene, ng/l	<44	<52	<41	760
Pyrene, ng/l	<8	<700	19000	14000

* The peaks for benzo-a-anthracene and chrysene co-elute -- the result given is the total result for these two compounds.

** The peaks for acenaphthene and fluorene co-elute -- the result given is the total result for these two compounds.

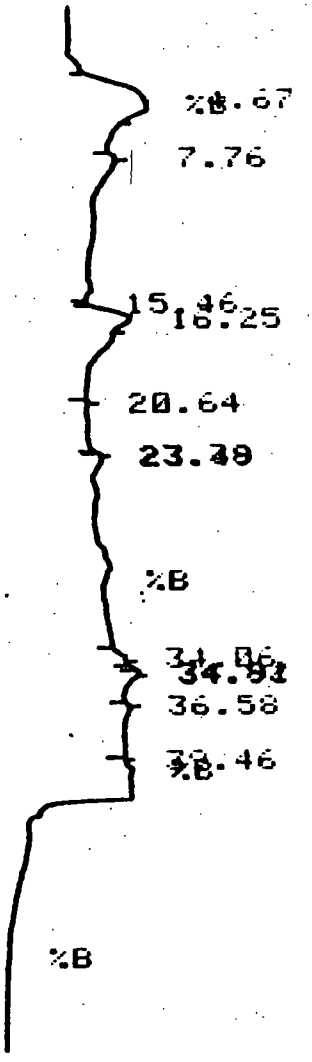
hp 1080 B

BTL: 3
ID:11-6-67726
ESTD FILE 1

RT	EXP RT	AREA		CAL #	AMT
17.52	18.03	38460		3	~~8181.11~~
23.52	23.70	3201	R	1	5.952
34.95	36.54	5092		8	~~9.706~~

DF: 1.6000 E+ 0

INJ START MESSAGE 8



Blank - 11/9/79

hp 1080 B

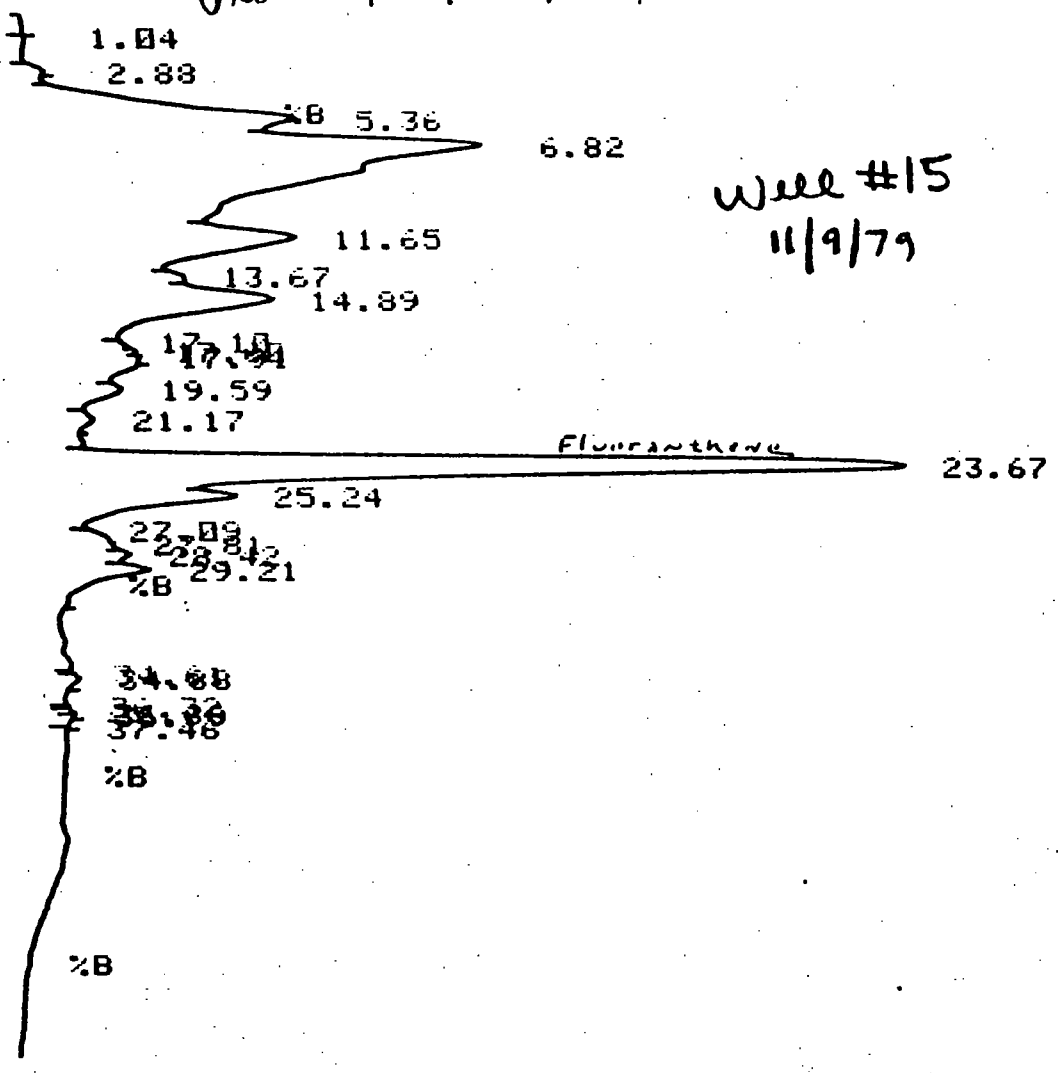
BTL: 4
ID:11-6-671.5
NO PEAKS IN WDOS

RT	AREA	AREA %
4.67	115300	115.825
16.25	41810	42.000
34.82	768	0.771
34.91	1397	1.403

DF: 1.6000 E+ 0

INJ START MESSAGE 8

```
ELETE CHG RUN @
HG RUN 2 OPTN   1 ID: 4 CLEAR #
  7 2 ESCAPE
HG RUN 2 OPTN   1 ID: 7 7 2 8 @
HG RUN 2 OPTN   4 DIL FACTOR: 1 . 5 @
HG RUN 3 OPTN   1 ID: 7 7 2 6 @
HG RUN 3 OPTN   4 DIL FACTOR: 1 . 6 @
HG RUN 4 OPTN   1 ID: 7 7 - 8 @
HG RUN 4 OPTN   1 ID: 1 . 5 @
HG RUN 5 OPTN   1 ID: 7 7 2 7 @
HG RUN 4 CLEAR #
  OPTN   4 DIL FACTOR: 1 . 9 @
HG RUN 6 OPTN   1 ID: . 0 1 A @
HG RUN 6 OPTN   4 DIL FACTOR: 1 @
HG RUN 6 STOP
MJ      START  MESSAGE 8th 1729 18 Volts
```



Well #15
11/9/79

```
    ** 1080 B

    BTL:    1
 D:11-6-66570
ESTD      FILE  1
```

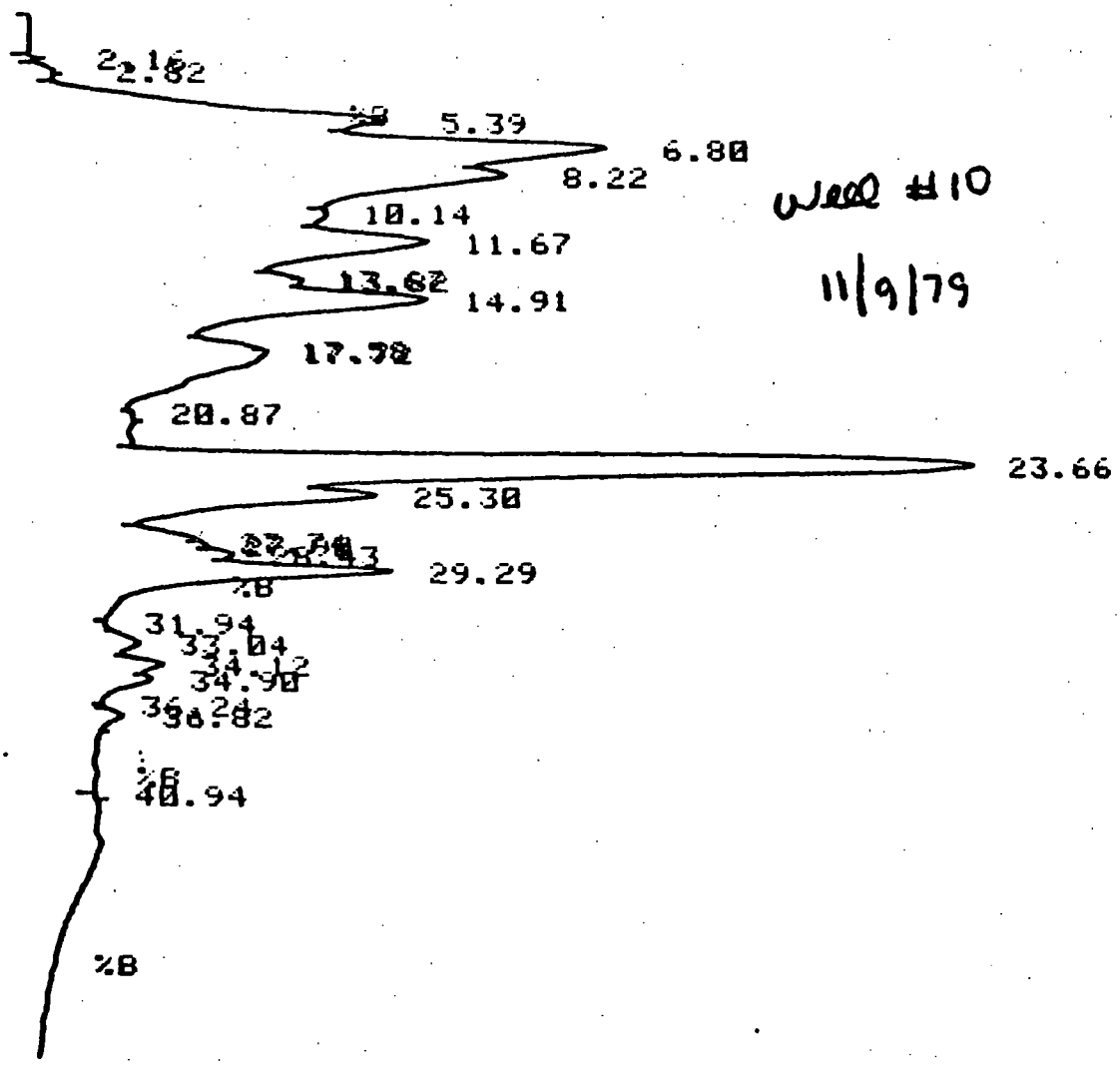
RT	EXP RT	AREA		CAL #	AMT
17.91	18.14	8678		3	~~3876.27~~
19.59	19.75	14490		4	421.478
21.17	21.13	12340		5	1331.66
23.67	23.70	1534000	R	1	1782.72
25.24	25.15	263100		6	7550.21
29.21	29.40	128600		7	486.109
36.79	36.77	1595		8	1.900

```
      DF:  1.0000 E+ 0
```

17.91	18.14	8678		3	~~3876.29~~
19.59	19.75	14490		4	421.478
21.17	21.13	12340		5	1331.66
23.67	23.70	1534000	R	1	1782.72
25.24	25.15	263100		6	7550.21
29.21	29.40	128600		7	486.109
36.79	36.77	1595		8	1.900

DF: 1.0000 E+ 0

INJ START MESSAGE 8



Well #10

11/9/79

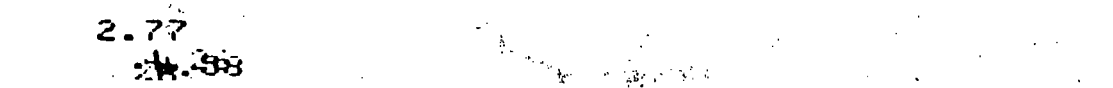
hp 1080 B

BTL: 2
ID:11-6-67728
ESTD FILE 1

RT	EXP RT	AREA		CAL #	AMT
17.72	18.13	443500		3	~~836026~~
23.66	23.70	1865000	R	1	3251.09
25.30	25.14	443300		6	19082.1
29.29	29.39	452700		7	2566.82
36.82	36.75	22000		8	39.314

DF: 1.5000 E+ 0

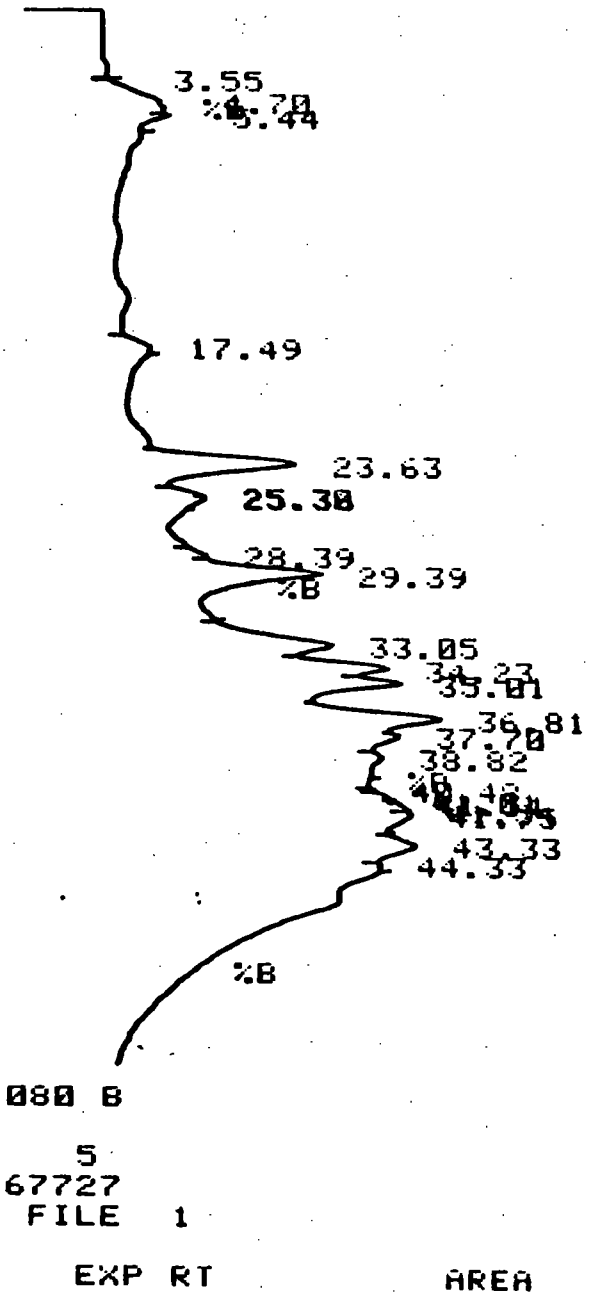
INJ START MESSAGE 8



10.23	4161U	42.000
34.82	768	0.771
34,91	1397	1.403

DF: 1.6000 E+ 0

INJ START MESSAGE 8



Well #9
11/9/79

hp 1080 B

BTL: 5
ID:11-6-67727
ESTD FILE 1

RT	EXP RT	AREA		CAL #	AMT
17.49	~~18.11~~ NO	9000		3	~~6861.83~~
23.63	23.70 yes	184600	R	1	407.609
25.30	25.11 yes	12900		6	703.366
29.39	29.35	189600		7	1361.71
36.81	36.71	194500		8	440.256
38.82	38.81	45420		9	87.876
43.33	43.34	37980		10	157.884

DF: 1.9000 E+ 0

INJ START MESSAGE 8

0.47

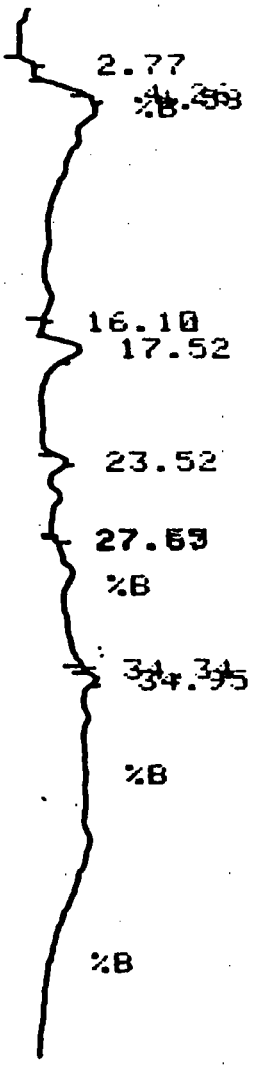
%B 1.44
6.30

BTL: 2
ID:11-6-67728
ESTD , FILE 1

RT	EXP RT	AREA		CAL #	AMT
17.72	18.13	443500		3	~~235826~~
23.66	23.70	1865000	R	1	3251.09
25.30	25.14	443300		6	19082.1
29.29	29.39	452700		7	2566.82
36.82	36.75	22000		8	39.314

DF: 1.5000 E+ 0

INJ START MESSAGE 8



Well #7
11/9/79

hp 1080 B

BTL: 3
ID:11-6-67726
ESTD FILE 1

RT	EXP RT	AREA		CAL #	AMT
17.52	18.03	38460		3	~~31814.1~~
23.52	23.70	3201	R	1	5.952
34.95	36.54	5092		8	~~9.706~~

DF: 1.6000 E+ 0

INJ START MESSAGE 8

APPENDIX F

MISCELLANEOUS

Adsorption On Activated Carbon

One of the potentially useful processes for control of toxic organic compounds is adsorption on activated carbon. It has the merit that the compounds are destroyed when the carbon is thermally regenerated.

Since adsorption is basically a surface phenomenon, it follows that substances with very high surface areas are desirable. Carbon is unique in that it possesses a very high surface area to mass ratio. Surface areas of carbons range from ~ 600 m^2/g to > 2000 m^2/g. Most of the surface area (95%) is contained in internal surfaces of pores and capillaries that are developed during activation of the carbon. An electron microscopic view of a carbon granule, shown in Figure 16 (provided by Dr. W. J. Weber of the University of Michigan) shows a rough exterior surface which is pockmarked by *holes* which constitute most of the surface area.

Adsorption is a complex process which involves both the nature of the carbon surface as well as the characteristic of the molecule. Some of the latter factors include solubility, molecular weight, polarity, ionization, orientation at the surface, and more. All of these factors contribute to the adsorbability of a compound which, operationally, can be determined quantitatively by determining a batch equilibrium adsorption isotherm, which mathematically can be described by the equation:

$$\frac{X}{M} = KC_f^{1/n}$$

where:

$\frac{X}{M}$ = the "loading" of the compound on the carbon in mg/g of carbon

C_f = the amount of compound remaining in solution after carbon treatment

K and 1/n are empirical constants.

The data, when plotted on log-log paper generally produces linear curves, from which much information can be inferred by observing the intercept, K, and the slope 1/n. Some typical isotherms are illustrated in Figure 17. It can be seen that adsorption of benzene, because of the steep slope of the isotherm, shows rapidly declining adsorption capacity on carbon and low residual concentrations would be difficult to achieve. The adsorption isotherms for β-naphthol and benzidine are more favorable for adsorption with the former being adsorbed at higher loadings on carbon. These isotherms were taken from a list of some 60 compounds that were collected into a single publication entitled *Carbon Adsorption Isotherms for Toxic Organics,* Municipal Environmental Research Laboratory, Cincinnati, Ohio 45268, May 1978.

While carbon is highly effective for the removal of organics, it is important to point out that the variation of loading on carbon is great. For example, the adsorption of 60 compounds mentioned above varied over 0-360 mg/g of carbon at an initial concentration of 1 mg/l of

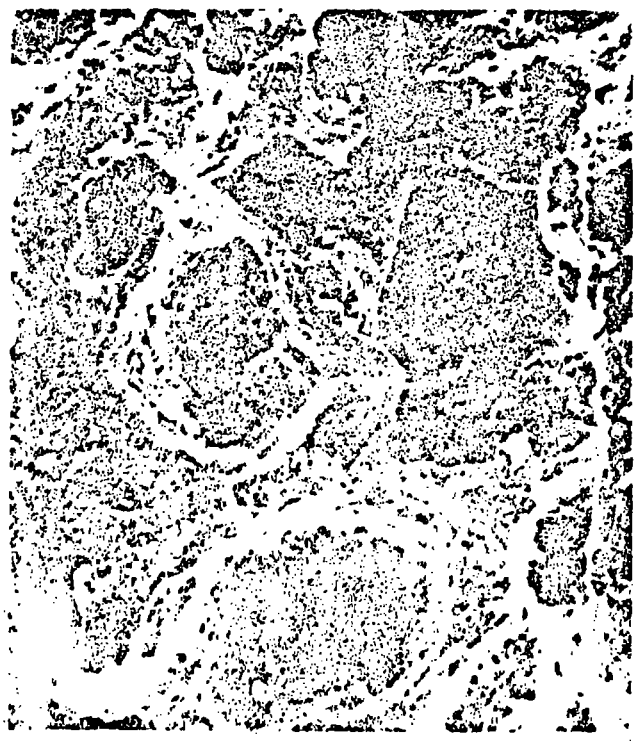


Figure 16. Electron Microphotograph of a Carbon Granule.

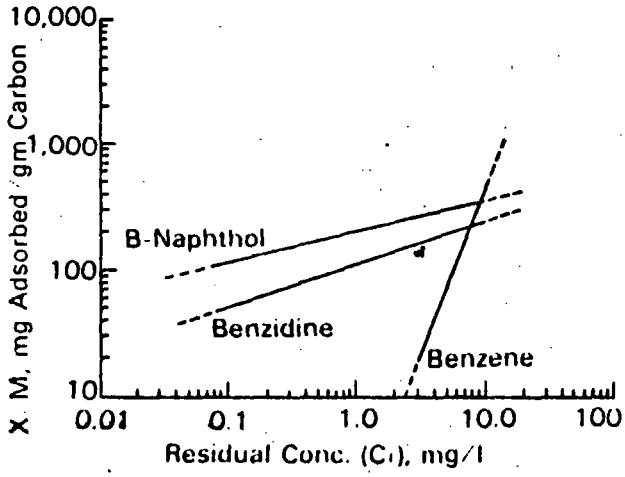
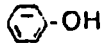
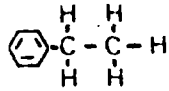
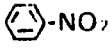
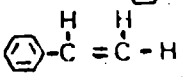
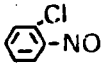


Figure 17. Carbon Adsorption Isotherms for Selected Compounds.

compound. Relatively small changes in a molecule can alter the adsorbability of the compound. This change in adsorbability is illustrated in Figure 18 which shows the marked effects of substitutions on a benzene molecule. All adsorption capacities are given at an initial concentration of compound of 1 mg/l. Thus, unsubstituted benzene is barely adsorbable, 0.7 mg/g. Substituting an OH group for one of the H in the benzene molecule increases the adsorption by a factor of 30. When Cl is substituted, this factor increases to 133.

In summary, adsorption (or non-adsorption) depends on many factors, not the least of which is the substitution on a parent molecule. It is one of the goals of the research on treatability to discover those factors which govern adsorption and ultimately to be able to predict adsorption in some systematic way. Without this capability, laboratories will be burdened with the need to evaluate adsorption for thousands of compounds.

Compound	Structure	Adsorption Capacity *(mg/g)
Benzene		0.7
Phenol		21
Ethylbenzene		53
Nitrobenzene		68
Chlorobenzene		93
Styrene		120
1-Chloro-2-Nitrobenzene		130

*Measured at 1 mg/l initial concentration

Figure 18. Adsorption Capacities for Benezene and Substituted Benezenes.

TECHNOLOGY DEVELOPMENT SUPPORT BRANCH

OBJECTIVES AND ACCOMPLISHMENTS

The Technology Development Support Branch provides technical and support services to the Division. It operates and maintains pilot plants and provides analytical services to all Division technology development operations. It is composed of the Pilot and Field Evaluation Section and the Waste Identification and Analysis Section.

Pilot and Field Evaluation Section

The Pilot and Field Evaluation Section is responsible for conducting most of the WRD pilot plant studies. These are conducted with U.S. EPA personnel at the Lebanon Pilot Plant and under contract with the Los Angeles County Sanitation Districts, Los Angeles County, California. In addition, personnel of this Section manage the national program in Instrumentation and Automation for Wastewater Treatment Systems. Some of the instrumentation and automation work is conducted at the pilot plant facilities mentioned above, but most is implemented through contracts and grants.

During this year a major activity was construction of the new Test and Evaluation Facility (Figure 19) on the grounds of the Mill Creek Sewage Treatment Plant in Cincinnati, Ohio. This 30,000 square foot facility will be the site of the major MERL pilot plant activity for many years in the future. The facility is equipped with all of the services required for research on water pollution control. Raw sewage, primary effluent, secondary effluent, primary sludge, secondary sludge, digested sludge and heat treatment liquor will be available on a continuous real time basis at any of 14 stations in the facility. In addition, access on a controlled basis to the industrial waste tank farm at this plant has been procured. This will make it possible to conduct studies on treatment of specific industrial wastes and on mixtures of industrial wastes and municipal wastes. The facility is equipped with a machine shop, dry chemical storage, wet chemical storage, two wet laboratories, an instrument laboratory, two cranes, office space, pure oxygen supply, compressed air supply, 110V, 220V, and 440V electric power. As much as possible, instrumentation will be used to monitor the processes under study. A computer system will be installed to log data, manipulate data, plot results, generate reports and implement process control.

Construction on this facility began in October 1977 and will be complete in January 1979. The facility should be operating at near full capacity by July 1979.

POLYNUCLEAR AROMATIC HYDROCARBONS

METHOD 610

1. Scope and Application

1.1 This method covers the determination of certain polynuclear aromatic hydrocarbons (PAH). The following parameters may be determined by this method:

Parameter	STORET No.	Parameter	STORET No.
Acenaphthene	34205	Chrysene	34320
Acenaphthylene	34200	Dibenzo(a,h)anthracene	34556
Anthracene	34220	Fluoranthene	34376
Benzo(a)anthracene	34526	Fluorene	34381
Benzo(a)pyrene	34247	Indeno(1,2,3-cd)pyrene	34403
Benzo(b)fluoranthene	34230	Naphthalene	34696
Benzo(ghi)perylene	34521	Phenanthrene	34461
Benzo(k)fluoranthene	34242	Pyrene	34469

1.2 This method is applicable to the determination of these compounds in municipal and industrial discharges. It is designed to be used to meet the monitoring requirements of the National Pollutant Discharge Elimination System (NPDES). As such, it presupposes a high expectation of finding the specific compounds of interest. If the user is attempting to screen samples for any or all of the compounds above, he must develop independent protocols for the verification of identity.

1.3 This method contains both liquid and gas chromatographic approaches, depending upon the needs of the analyst. The gas

chromatographic procedure cannot adequately resolve the following four pairs of compounds: anthracene and phenanthrene; chrysene and benzo(a)anthracene; benzo(b)fluoranthene and benzo(k)fluoranthene; and dibenzo(a,h)anthracene and indeno(1,2,3-cd)pyrene. Unless the purposes of the analysis can be served by reporting a sum for an unresolved pair, the liquid chromatographic approach must be used for these compounds. The liquid chromatographic method will resolve all of the 16 compounds listed above.

1.4 The sensitivity of this method is usually dependent upon the level of interferences rather than instrumental limitations. The limits of detection listed in Table I for the liquid chromatographic approach represent sensitivities that can be achieved in wastewaters in the absence of interferences.

1.5 This method is recommended for use only by experienced residue analysts or under the close supervision of such qualified persons.

2. Summary of Method

2.1 A 1-liter sample of wastewater is extracted with methylene chloride using separatory funnel techniques. The extract is dried and concentrated to a volume of 10 ml or less. Chromatographic conditions are described which allow for the accurate measurement of the compounds in the extract by either High Performance Liquid Chromatography (HPLC) or gas chromatography.

2.2 If interferences are encountered, the method provides a selected general purpose cleanup procedure to aid the analyst in their elimination.

3. Interferences

3.1 Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of the chromatograms. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required.

3.2 Interferences coextracted from the samples will vary considerably from source to source, depending upon the diversity of the industrial complex or municipality being sampled. While a general clean-up technique is provided as part of this method, unique samples may require additional clean-up approaches to achieve the sensitivities stated in Table 1.

3.3 The extent of interferences that may be encountered using liquid chromatographic techniques has not been fully assessed. Although the chromatographic conditions described allow for a unique resolution of the specific PAH compounds covered by this method, other PAH compounds may interfere.

4. Apparatus and Materials

4.1 Sampling equipment, for discrete or composite sampling.

4.1.1 Grab sample bottle - amber glass, 1-liter or 1-quart volume. French or Boston Round design is recommended. The container must be washed and solvent rinsed before use to minimize interferences.

4.1.2 Bottle caps - Threaded to screw on to the sample bottles. Caps must be lined with Teflon. Foil may be substituted if

sample is not corrosive.

4.1.3 Compositing equipment - Automatic or manual compositing system. Must incorporate glass sample containers for the collection of a minimum of 250 ml. Sample containers must be kept refrigerated during sampling. No tygon or rubber tubing may be used in the system.

4.2 Separatory funnel - 2000 ml, with Teflon stopcock.

4.3 Drying column - 20 mm ID pyrex chromatographic column with coarse frit.

4.4 Kuderna-Danish (K-D) Apparatus

4.4.1 Concentrator tube - 10 ml, graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked. Ground glass stopper (size 19/22 joint) is used to prevent evaporation of extracts.

4.4.2 Evaporative flask - 500 ml (Kontes K-57001-0500 or equivalent). Attach to concentrator tube with springs. (Kontes K-662750-0012).

4.4.3 Snyder column - three-ball macro (Kontes K503000-0121 or equivalent).

4.4.4 Snyder column - two-ball micro (Kontes K-569001-0219 or equivalent).

4.4.5 Boiling chips - solvent extracted, approximately 10/40 mesh.

4.5 Water bath - Heated, with concentric ring cover, capable of temperature control ($\pm$ 2°C). The bath should be used in a hood.

4.6 HPLC Apparatus:

4.6.1 Gradient pumping system, constant flow.

4.6.2 Reverse phase column, 5 micron HC-ODS Sil-X, 250 mm x 2.6 mm ID (Perkin Elmer No. 809-0716 or equivalent).

4.6.3 Fluorescence detector, for excitation at 280 nm and emission at 389 nm.

4.6.4 UV detector, 254 nm, coupled to fluorescence detector.

4.6.5 Strip chart recorder compatible with detectors, (A data system for measuring peak areas is recommended).

4.7 Gas chromatograph - Analytical system complete with gas chromatograph suitable for on-column injection and all required accessories including dual flame ionization detectors, column supplies, recorder, gases, syringes. A data system for measuring peak areas is recommended.

4.8 Chromatographic column - 250 mm long x 10 mm ID with coarse fritted disc at bottom and Teflon stopcock.

5. Reagents

5.1 Preservatives:

5.1.1 Sodium hydroxide - (ACS) 10 N in distilled water.

5.1.2 Sulfuric acid - (ACS) Mix equal volumes of conc. H_2SO_4 with distilled water.

5.1.3 Sodium thiosulfate - (ACS) Granular.

5.2 Methylene chloride, Pentane, Cyclohexane, High Purity Water-HPLC quality, distilled in glass.

5.3 Sodium sulfate - (ACS) Granular, anhydrous (purified by heating at 400°C for 4 hrs. in a shallow tray).

5.4 Stock standards - Prepare stock standard solutions at a concentration of 1.00 ug/ul by dissolving 0.100 grams of assayed

reference material in pesticide quality isooctane or other appropriate solvent and diluting to volume in a 100 ml ground glass stoppered volumetric flask. The stock solution is transferred to ground glass stoppered reagent bottles, stored in a refrigerator, and checked frequently for signs of degradation or evaporation, especially just prior to preparing working standards from them.

5.5 Acetonitrile - Spectral quality.

5.6 Silica gel - 100/120 mesh desiccant (Davison Chemical grade 923 or equivalent). Before use, activate for at least 16 hours at 130°C in a foil covered glass container.

6. Calibration

6.1 Prepare calibration standards that contain the compounds of interest, either singly or mixed together. The standards should be prepared at concentrations covering two or more orders of-magnitude that will completely bracket the working range of the chromatographic system. If the sensitivity of the detection system can be calculated from Table I as 100 ug/l in the final extract, for example, prepare standards at 10 ug/l, 50 ug/l, 100 ug/l, 500 ug/l, etc. so that injections of 1-5 ul of each calibration standard will define the linearity of the detector in the working range.

6.2 Assemble the necessary HPLC or gas chromatographic apparatus and establish operating parameters equivalent to those indicated in Table I or II. By injecting calibration standards, establish the sensitivity limit of the detectors and the linear range of the analytical systems for each compound.

6.3 Before using any cleanup procedure, the analyst must process a series of calibration standards through the procedure to validate elution patterns and the absence of interferences from the reagents.

7. Quality Control

7.1 Before processing any samples, the analyst should demonstrate through the analysis of a distilled water method blank, that all glassware and reagents are interference-free. Each time a set of samples is extracted or there is a change in reagents, a method blank should be processed as a safeguard against chronic laboratory contamination.

7.2 Standard quality assurance practices should be used with this method. Field replicates should be collected to validate the precision of the sampling technique. Laboratory replicates should be analyzed to validate the precision of the analysis. Fortified samples should be analyzed to validate the accuracy of the analysis. Where doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as fraction collection and GC-mass spectroscopy should be used.

8. Sample Collection, Preservation, and Handling

8.1 Grab samples must be collected in glass containers. Conventional sampling practices should be followed, except that the bottle must not be prewashed with sample before collection. Composite samples should be collected in refrigerated glass containers in accordance with the requirements of the program. Automatic sampling equipment must be free of tygon and other potential sources of contamination.

8.2 The samples must be iced or refrigerated from the time of

collection until extraction. Chemical preservatives should not be used in the field unless more than 24 hours will elapse before delivery to the laboratory. If the samples will not be extracted within 48 hours of collection, adjust the sample to a pH range of 6.0-8.0 with sodium hydroxide or sulfuric acid and add 35 mg sodium thiosulfate per part per million of free chlorine per liter.

8.3 All samples must be extracted within 7 days and completely analyzed within 30 days of collection.

9. Sample Extraction

9.1 Mark the water meniscus on the side of the sample bottle for later determination of sample volume. Pour the entire sample into a two-liter separatory funnel. Check the pH of the sample with wide-range pH paper and adjust to within the range of 5-9 with sodium hydroxide or sulfuric acid.

9.2 Add 60 ml methylene chloride to the sample bottle, seal, and shake 30 seconds to rinse the inner walls. Transfer the solvent into the separatory funnel, and extract the sample by shaking the funnel for two minutes with periodic venting to release vapor pressure. Allow the organic layer to separate from the water phase for a minimum of ten minutes. If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration of the emulsion through glass wool, or centrifugation. Collect the methylene chloride extract in a 250-ml Erlenmeyer flask.

9.3 Add a second 60-ml volume of methylene chloride to the sample bottle and complete the extraction procedure a second time, combining the extracts in the Erlenmeyer flask.

9.4 Perform a third extraction in the same manner. Pour the combined extract through a drying column containing 3-4 inches of anhydrous sodium sulfate, and collect it in a 500-ml Kuderna-Danish (K-D) flask equipped with a 10 ml concentrator tube. Rinse the Erlenmeyer flask and column with 20-30 ml methylene chloride to complete the quantitative transfer.

9.5 Add 1-2 clean boiling chips to the flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1 ml methylene chloride to the top. Place the K-D apparatus on a hot water bath (60-65°C) so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed in vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15-20 minutes. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood. When the apparent volume of liquid reaches 1 ml, remove the K-D apparatus and allow it to drain for at least 10 minutes while cooling. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1-2 ml of methylene chloride. A 5-ml syringe is recommended for this operation. Stopper the concentrator tube and store refrigerated if further processing will not be performed immediately.

9.6 Determine the original sample volume by refilling the sample bottle

to the mark and transferring the liquid to a 1000 ml graduated cylinder. Record the sample volume to the nearest 5 ml.

9.7 If the sample requires cleanup before chromatographic analysis, proceed to Section 10. If the sample does not require cleanup, or if the need for cleanup is unknown, analyze an aliquot of the extract according to Section 11 or Section 12.

10. Cleanup and Separation

10.1 Before the silica gel cleanup technique can be utilized, the extract solvent must be exchanged to cyclohexane. Add a 1-10 ml aliquot of sample extract (in methylene chloride) and a boiling chip to a clean K-D concentrator tube. Add 4 ml cyclohexane and attach a micro-Snyder column. Prewet the micro-Snyder column by adding 0.5 ml methylene chloride to the top. Place the micro-K-D apparatus on a boiling (100°C) water bath so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete concentration in 5-10 minutes. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood. When the apparent volume of the liquid reaches 0.5 ml, remove the K-D apparatus and allow it to drain for at least 10 minutes while cooling. Remove the micro-Snyder column and rinse its lower joint into the concentrator tube with a minimum of cyclohexane. Adjust the extract volume to about 2 ml.

10.2 Silica Gel Column Cleanup for PAHs.

10.2.1 Prepare a slurry of 10g activated silica gel in methylene

chloride and place this in a 10 mm ID chromatography column. Gently tap the column to settle the silica gel and elute the methylene chloride. Add 1-2 cm of anhydrous sodium sulfate to the top of the silica gel.

10.2.2 Preelute the column with 40 ml pentane. Discard the eluate and just prior to exposure of the sodium sulfate layer to the air, transfer the 2 ml cyclohexane sample extract onto the column, using an additional 2 ml of cyclohexane to complete the transfer.

10.2.3 Just prior to exposure of the sodium sulfate layer to the air, add 25 ml pentane and continue elution of the column. Discard the pentane eluate.

10.2.4 Elute the column with 25 ml of 40% methylene chloride/60% pentane and collect the eluate in a 500 ml K-D flask equipped with a 10 ml concentrator tube. Elution of the column should be at a rate of about 2 ml/min.

10.2.5 Concentrate the collected fraction to less than 10 ml by K-D techniques as in 9.5, using pentane to rinse the walls of the glassware. Proceed with HPLC or gas chromatographic analysis.

11. High Performance Liquid Chromatography HPLC

11.1 To the extract in the concentrator tube, add 4 ml acetonitrile and a new boiling chip, then attach a micro-Snyder column. Increase the temperature of the hot water bath to 95-100°C. Concentrate the solvent as above. After cooling, remove the micro-Snyder column and rinse its lower joint into the concentrator tube with

about 0.2 ml acetonitrile. Adjust the extract volume to 1.0 ml.

11.2 Table I summarizes the recommended HPLC column materials and operating conditions for the instrument. Included in this table are estimated retention times and sensitivities that should be achieved by this method. An example of the separation achieved by this column is shown in Figure 1. Calibrate the system daily with a minimum of three injections of calibration standards.

11.3 Inject 2-5 ul of the sample extract with a high pressure syringe or sample injection loop. Record the volume injected to the nearest 0.05 ul, and the resulting peak size, in area units.

11.4 If the peak area exceeds the linear range of the system, dilute the extract and reanalyze.

11.5 If the peak area measurement is prevented by the presence of interferences, further cleanup is required.

11.6 The UV detector is recommended for the determination of naphthalene and acenaphthylene and the fluorescence detector is recommended for the remaining PAHs.

12. Gas Chromatography

12.1 The gas chromatographic procedure will not resolve certain isomeric pairs as indicated in Table II. The liquid chromatographic procedure (Section 11) must be used for these materials.

12.2 To achieve maximum sensitivity with this method, the extract must be concentrated to 1.0 ml. Add a clean boiling chip to the methylene chloride extract in the concentrator tube. Attach a two-ball micro-Snyder column. Prewet the micro-Snyder column by adding about 0.5 ml of methylene chloride to the top. Place this

micro-K-D apparatus on a hot water bath (60-65°C) so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and water temperature as required to complete the concentration in 5 to 10 minutes. At the proper rate of distillation the balls will actively chatter but the chambers will not flood. When the apparent volume of liquid reaches 0.5 ml, remove the K-D apparatus and allow it to drain for at least 10 minutes while cooling. Remove the micro-Snyder column and rinse its lower joint into the concentrator tube with a small volume of methylene chloride. Adjust the final volume to 1.0 ml and stopper the concentrator tube.

12.3 Table II describes the recommended gas chromatographic column material and operating conditions for the instrument. Included in this table are estimated retention times that should be achieved by this method. Calibrate the gas chromatographic system daily with a minimum of three injections of calibration standards.

12.4 Inject 2-5 ul of the sample extract using the solvent-flush technique. Smaller (1.0 ul) volumes can be injected if automatic devices are employed. Record the volume injected to the nearest 0.05 ul, and the resulting peak size, in area units.

12.5 If the peak area exceeds the linear range of the system, dilute the extract and reanalyze.

12.6 If the peak area measurement is prevented by the presence of interferences, further cleanup is required.

13. Calculations

13.1 Determine the concentration of individual compounds according to

the formula:

$$\text{Concentration, ug/l} = \frac{(A)\ (B)\ (V_t)}{(V_i)\ \ (V_s)}$$

where A = Calibration factor for chromatographic system, in nanograms material per area unit.

B = Peak size in injection of sample extract, in area units

V_i = volume of extract injected (ul)

V_t = Volume of total extract (ul)

V_s = Volume of water extracted (ml)

13.2 Report results in micrograms per liter without correction for recovery data. When duplicate and spiked samples are analyzed, all data obtained should be reported.

14. Accuracy and Precision

14.1 The U.S. EPA Environmental Monitoring and Support Laboratory in Cincinnati is in the process of conducting an interlaboratory method study to determine the accuracy and precision of this test procedure.

BIBLIOGRAPHY

"Development and Application of Test Procedures for Specific Organic Toxic Substances in Wastewaters, Category 9-PAHs." Report for EPA Contract 68-03-2624 (In preparation).

TABLE I

High Performance Liquid Chromatography of PAH's

Compound	Retention Time (min)	Detection Limit (ug/l) UV	Fluorescence
Naphthalene	16.17	2.5	20.0
Acenaphthylene	18.10	5.0	100.0
Acenaphthene	20.14	3.0	4.0
Fluorene	20.89	0.5	2.0
Phenanthrene	22.32	0.25	1.2
Anthracene	23.78	0.10	1.5
Fluoranthene	25.00	0.50	0.05
Pyrene	25.94	0.10	0.05
Benzo(a)anthracene	29.26	0.20	0.04
Chrysene	30.14	0.20	0.5
Benzo(b)fluoranthene	32.44	1.0	0.04
Benzo(k)fluoranthene	33.91	0.30	0.04
Benzo(a)pyrene	34.95	0.25	0.04
Dibenzo(a,h)anthracene	37.06	1.0	0.08
Benzo(ghi)perylene	37.82	0.75	0.2
Indeno(1,2,3-cd)pyrene	39.21	0.30	0.1

HPLC conditions: Reverse phase HC-ODS Sil-X 2.6 x 250 mm Perkin-Elmer column; isocratic elution for 5 min. using 40% acetonitrile/60% water, then linear gradient elution to 100% acetonitrile over 25 minutes; flow rate is 0.5 ml/min.

Detection limit is calculated from the minimum detectable HPLC response being equal to five times the background noise, assuming an equivalent of a 2 ml final volume of the 1 liter sample extract, and assuming an HPLC injection of 2 microliters.

TABLE II

Gas Chromatography of PAHs

Compound	Retention Time (Min)
Naphthalene	4.5
Acenaphthylene	10.4
Acenaphthene	10.8
Fluorene	12.6
Phenanthrene	15.9
Anthracene	15.9
Fluoranthene	19.8
Pyrene	20.6
Benzo(a)anthracene	20.6
Chrysene	24.7
Benzo(b)fluoranthene	28.0
Benzo(k)fluoranthene	28.0
Benzo(a)pyrene	29.4
Dibenzo(a,h)anthracene	36.2
Indeno(1,2,3-cd)pyrene	36.2
Benzo(ghi)perylene	38.6

GC conditions: Chromosorb W-AW-DCMS 100/120 mesh coated with 3% OV-17, packed in a 6' x 2 mm ID glass column, with nitrogen carrier gas at 40 ml/min flow rate. Column temperature was held at 100°C for 4 minutes, then programmed at 8°/minute to a final hold at 280°C.

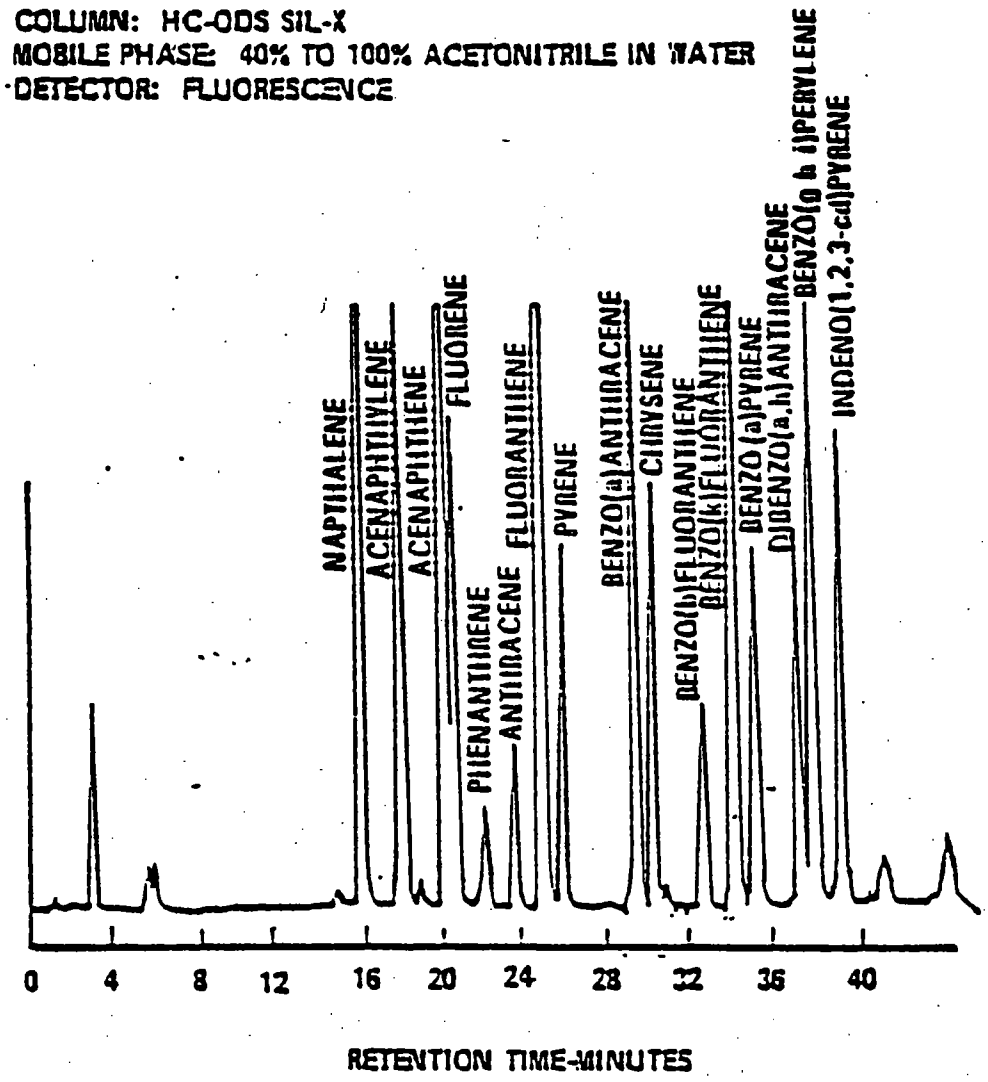


Figure 1. Liquid chromatogram of polynuclear aromatics

minnesota department of health
717 s.e. delaware st. minneapolis 55440
(612) 296-5221

September 5, 1979

Mr. Vern Tollefsrud
Water Superintendent
City of St. Louis Park
5005 Minnetonka Boulevard
St. Louis Park, Minnesota 55416

Dear Vern:

Enclosed are the polynuclear aromatic hydrocarbon results from the charcoal study conducted during the week of July 16th, 1979. A decrease in concentration of various PAH compounds is seen at the 3 ppm charcoal level and at the 14 ppm (average) charcoal level. Because of the erratic pump behavior at the higher charcoal level, I would recommend repeating the test at 6 ppm and 12 ppm charcoal levels to verify effective removal of PAH compounds over time.

Sincerely yours,

William Scruton

William Scruton
Research Scientist II
Section of Health Risk Assessment

WS:jm
Enclosure

ase Fill Out
Triplicate

SPECIAL SAMPLE DATA SHEET

Page 1 of 1

Collected by Vern Tallman

Date Collected 7/16+7/17

Report to Bill Semrau - IRA

Program Element # 214

MDH Coordinator JMC

Expected Compl. Date open

Date Rec'd By Lab 7/17/79

Lab. Sample # 5005-77

SPECIAL SAMPLE DESCRIPTION:

✓ Water ___ Sediment ___ Sludge ___ Fish ___ Other (Specify)

SAMPLE NO.	FIELD NO.	SAMPLING POINT OR SOURCE	TYPE OF BOTTLES REC'D**	Tot. No. EACH
a. 5005	3	Well 15, water → sand filter	(11 am) 4 ℓ	1
b. 5006	4	" , water → sand filter	(11 PM) "	1
c. 5007	5	" , "	(5 PM) "	1
d.				
e.				
f.				

SPECIAL SAMPLE ANALYSES REQUESTED: *. HPLC Scan charcoal started at 9ca PM 7/16/79

COMPLETED AUG 27 1979

OTHER ANALYSES REQUESTED ALSO AND WILL BE REPORTED SEPARATELY FROM SPECIAL SAMP. ANALYSES

VICAL SERVICE

SPECIAL INSTRUCTIONS AND COMMENTS:

RESULTS:

ONLY

ease Fill Out
Triplicate

SPECIAL SAMPLE DATA SHEET

Collected by Vern Tallefson

MDH Coordinator [illegible]

Date Collected 7/26-27

Expected Compl. Date [illegible]

Report to Bill Semrau-MAA

Date Rec'd By Lab

Program Element # 214

Lab. Sample # 55089-92

SPECIAL SAMPLE DESCRIPTION:

X Water ___ Sediment ___ Sludge ___ Fish ___ Other (Specify)

SAMPLE NO.	FIELD NO.	SAMPLING POINT OR SOURCE	TYPE OF BOTTLES REC'D**	Tot. No. EACH
a. [illegible]	1	[illegible]	4ℓ	
b. [illegible]	[illegible]	[illegible]		
c. [illegible]	7	duplicate of 6		
d. [illegible]	[illegible]	[illegible]		
e. [illegible]	[illegible]	[illegible]		
f. [illegible]	10	duplicate of [illegible]	✓	

ECIAL SAMPLE
ALYSES REQUESTED: * HPLC Scan

COMPLETED

AUG 27 1979

[illegible]TICAL SERVICE

OTHER ANALYSES REQUESTED ALSO AND WILL BE REPORTED SEPARATELY FROM SPECIAL SAMP. ANALYSES

ECIAL INSTRUCTIONS AND COMMENTS:

ULTS:

Please Fill Out
Triplicate

SPECIAL SAMPLE DATA SHEET

Collected by Vern Tallman

Date Collected 7/31-8/1

Report to Bill Sommers, MDH

Program Element # 2.7

MDH Coordinator JMK

Expected Compl. Date 9/1/79

Date Rec'd By Lab 8/1/79

Lab. Sample # 55073

SPECIAL SAMPLE DESCRIPTION:

X Water ___ Sediment ___ Sludge ___ Fish ___ Other (Specify)

SAMPLE NO.	FIELD NO.	SAMPLING POINT OR SOURCE	TYPE OF BOTTLES REC'D**	Tot. No. EACH
a. 55073	11	Well - Farm	Quart 4X	1
b.				
c.				
d.				
e.				
f.				

SPECIAL SAMPLE ANALYSES REQUESTED: * HLC Scan

COMPLETED

AUG 27 1979

OTHER ANALYSES REQUESTED ALSO AND WILL BE REPORTED SEPARATELY FROM SPECIAL SAMP. ANALYSES

SPECIAL INSTRUCTIONS AND COMMENTS:

RESULTS:

** List Bottles Received for Special Sample Analyses ONLY.

ease Fill Out Triplicate

SPECIAL SAMPLE DATA SHEET

Collected by [illegible]

Date Collected 7/18-19

Report to Bill S[illegible]

Program Element # 211

MDH Coordinator [illegible]

Expected Compl. Date [illegible]

Date Rec'd By Lab 7/19/79

Lab. Sample # 5[illegible]

SPECIAL SAMPLE DESCRIPTION:

X Water ___Sediment ___Sludge ___Fish ___Other (Specify)

SAMPLE NO.	FIELD NO.	SAMPLING POINT OR SOURCE	TYPE OF BOTTLES REC'D**	Tot. No. EACH
a.	3	[illegible]	ℓ	1
b.	5	[illegible]		
c.	4	[illegible]		
d.	12	[illegible]		
e.		Background [illegible]		
f.	14	[illegible]	1	

SPECIAL SAMPLE ANALYSES REQUESTED: * HPLC Scan

COMPLETED

AUG 27 1979

OTHER ANALYSES REQUESTED ALSO AND WILL BE REPORTED SEPARATELY FROM SPECIAL SAMP. ANALYSES

SPECIAL INSTRUCTIONS AND COMMENTS:

RESULTS:

1st Bottles Received for Special Sample Analyses ONLY.

rlease Fill Out
rn Triplicate

SPECIAL SAMPLE DATA SHEET

Collected by [illegible]

Date Collected 7/[illegible]/79

Report to Bill S[illegible]

Program Element # 214

MDH Coordinator [illegible]

Expected Compl. Date [illegible]

Date Rec'd By Lab 7/19/79

Lab. Sample # [illegible]

SPECIAL SAMPLE DESCRIPTION:

X Water ___Sediment ___Sludge ___Fish ___Other (Specify)

SAMPLE NO.	FIELD NO.	SAMPLING POINT OR SOURCE	TYPE OF BOTTLES REC'D**	Tot. No. EACH
a. [illegible]	15	[illegible]	4l	1
b. [illegible]	2	[illegible]	↓	↓
c. [illegible]	16	[illegible]	(2nd test) ↓	↓
d.				
e.				
f.				

SPECIAL SAMPLE NALYSES REQUESTED: * HPLC Scan

COMPLETED

AUG 27 1979

___ OTHER ANALYSES REQUESTED ALSO AND WILL BE REPORTED SEPARATELY FROM SPECIAL SAMP. ANALYSES

SPECIAL INSTRUCTIONS AND COMMENTS:

RESULTS:

* List Bottles Received for Special Sample Analyses ONLY.

Please Fill Out
In Triplicate

SPECIAL SAMPLE DATA SHEET

Page 1 of 1

Collected by [illegible]

MDH Coordinator ACT

Date Collected 7/19-20

Expected Compl. Date ASAP

Report to Bill [illegible] - HRA

Date Rec'd By Lab 7/20/79

Program Element # 2[illegible]

Lab. Sample # 5[illegible]

SPECIAL SAMPLE DESCRIPTION:

X Water ___ Sediment ___ Sludge ___ Fish ___ Other (Specify)

SAMPLE NO.	FIELD NO.	SAMPLING POINT OR SOURCE	TYPE OF BOTTLES REC'D**	Tot. No. EACH
a. 55111	1	Well #5 - Filter [illegible]	[illegible] 4l	1
b. [illegible]	6	SC 7/[illegible]	[illegible]	
c. [illegible]	7	11 C 7/[illegible]	[illegible]	
d. [illegible]	8	5 RO 7/2[illegible]	[illegible]	
e. [illegible]	17	Raw water SC 7/2[illegible]	[illegible]	
f.				

SPECIAL SAMPLE ANALYSES REQUESTED: * 111LC Scan

COMPLETED

AUG 27 1979

[illegible]TICAL SERVICES

OTHER ANALYSES REQUESTED ALSO AND WILL BE REPORTED SEPARATELY FROM SPECIAL SAMP. ANALYSES

SPECIAL INSTRUCTIONS AND COMMENTS:

RESULTS:

List Bottles Received for Special Sample Analyses ONLY.

Concentration of Selected P.A.H. Compounds (ng/l)

COMPOUND	7/19 5 p.m. 55103 *	7/19 11:00 p.m. 55119 *	7/19 5 p.m. 55120 *	7/19 11 a.m. 55121 *	7/20 5 a.m. 55122 *	Results	
2-Methylnaphthalene	<6.7.	---	---	---	---	SAME	
Acenaphthene ✓	110.	21.	<2.2	<2.2	<2.2	3200 -	<2.2
Biphenyl	240.	59.	6.5	9.5	3.6	450 -	3.6 -
Anthracene	<8.0	<8.0	<8.0	<8.0	<8.0	600 -	<8.0
Phenanthrene	510.	10.	27.	<1.0	<1.0	1900-1700 -	<1.0
Pyrene	***	***	***	***	***	—	
1,2,3,7-tetrahydro-pyrene							
Fluorene						510 - 190	
Fluoranthene	14.	7.0	3.6	0.99	1.2	~~Benzene~~ 700	- 14. - 1.2 - 0.99
1,2-Benzanthracene	<52.	---	---	---	---	?	
Chrysene	<25.	---	---	---	---	SAME ?	
Benzo (a) pyrene	<2.2	<2.2	<2.2	<2.2	<2.2	SAME	
3,4-Benzphenanthrene	<1.0	<1.0	<1.0	<1.0	<1.0	<15. - <1.0	
Benzo (e) pyrene							
Perylene	<1.0	<1.0	<1.0	<1.0	<1.0	SAME	
Benzo (g,h,i) perylene							
Benzo (k) fluoranthene							
Benzo (j) fluoranthene							
Benzo (b) fluoranthene							
2-phenylenepyrene	<1.0	<1.0	<1.0	<1.0	<1.0	SAME	
1,2-5,6-Dibenzan-thracene	<1.0	<1.0	<1.0	<1.0	<1.0	38. -	.0

... column and data system installed on 7/21/79. Acenaphthene and Biphenyl no longer co-elute.
... and Biphenyl co-eluted. The peak was calculated as Acenaphthene.
... and Pyrene co-eluted. The peak was calculated as Phenanthrene.
... not in standard.

Filter effluent treated

Concentration of Selected P.A.H. Compounds (ng/l)

COMPOUND	7/17 11:00 A.M. 55088	7/17 11:00 AM 55089	7/17 5:00 PM 55090	7/17 11:00 P.M. + 55091	7/18 11:00 A.M. 55092	7/18 5:00 P.M. 55093	Filter influent 7/18 11:00 A.M. 55096	7/18 5:00 P.M. + 55097	7/18 9:00 P.M. + 55098
2-Methylnaphthalene	<67.	<67.	<67.	<67.	<67.	<67.	<67.	---	---
Acenaphthene ✓	910.	1200.	1300.	170.	2700.	1500.	2600.	190.	190.
Diphenyl	* *	* *	* *	350.	* *	* *	* *	380.	410.
Anthracene ✓				<8.0				<8.0	<8.0
Phenanthrene ✓	430.	360.	300.	180.	190.	180.	210.	210.	240.
Pyrene ✓	* * *	* * *	* * *	* * *	* * *	* * *	* * *	* * *	* * *
1,2,6,7-tetrahydro-pyrene									
Fluorene ✓									
Fluoranthene ✓	39.	81.	62	39.	30.	28.	41.	17.	15.
3,4-Benzanthracene	<52.	<52.	<52.	<52.	<52.	<52.	<52	---	---
Chrysene ✓	<25.	<25.	<25.	<25.	<25.	<25.	<25.	---	---
Benzo (e) pyrene	<2.2	<2.2	<2.2	<2.2	<2.2	<2.2	<2.2	<2.2	<2.2
3,4-Benzphenanthrene	<15.	<15.	<15.	<1.0	<15.	<15.	<15.	<1.0	<1.0
Benzo (a) pyrene									
Perylene	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
Benzo (g,h,i) perylene									
Benzo (k) fluoranthene									
Benzo (j) fluoranthene									
Benzo (b) fluoranthene									
3-phenylenepyrene	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
1,2-5,6-Dibenzan-thracene	<38.	<38.	<38.	<1.0	<38.	<38.	<38.	<38	<38.

* Column and data system installed on 7/20/79. Acenaphthene and Diphenyl no longer co-elute.
** Acenaphthene and Diphenyl co-eluted. The peak was calculated as Acenaphthene.
*** Phenanthrene and Pyrene co-eluted. The peak was calculated as Phenanthrene.
--- Compound not in standard.

7/16 DSAN 11:05 AM F. ITR. NO Treatment

Concentration of Selected P.A.H. Compounds (ng/l)

COMPOUND	55075	55076	55077	55100	55101				
		7/16 11:00 PM	7/16 6:00 AM	7/19 3:00 AM	Dup-3:00 AM 7/19				
-Methylnaphthalene	<67.	<67.	<67.	<67.	<67.				
Acenaphthene	<140.	2500.	1600.	4800.	3700.				
Biphenyl	* *	* *	* *	* *	* *				
Anthracene									
Phenanthrene	6.1	1900.	440.	1100.	1000.				
Pyrene	* * *	* * *	* * *	* * *	* * *				
1,2,3,7-tetrahydro-pyrene									
Fluorene									
Fluoranthene	3.6	84.	91.	32.	32.				
1,2-Benzanthracene	<52.	<52.	<52.	<52.	<52.				
Chrysene	<25.	<25.	<25.	<25.	<25.				
Benzo (e) pyrene	<2.2	<2.2	<2.2	<2.2	<2.2				
3,4-Benzphenanthrene	<15.	<15.	<15.	<15.	<15.				
Benzo (a) pyrene									
Perylene	<1.0	<1.0	<1.0	<1.0	<1.0				
Benzo (g,h,i) perylene									
Benzo (k) fluoranthene									
Benzo (j) fluoranthene									
Benzo (b) fluoranthene									
1,2-phenylenepyrene	<1.0	<1.0	<1.0	<1.0	<1.0				
Anthracene-1,2-5,6-Dibenzan-thracene	<38.	<38.	<38.	<38.	<38.				

New column and data system installed on 7/26/79. Acenaphthene and Biphenyl no longer co-elute.
* Acenaphthene and Biphenyl co-eluted. The peak was calculated as Acenaphthene.
** Phenanthrene and Pyrene co-eluted. The peak was calculated as Phenanthrene.
--- compound not in standard.

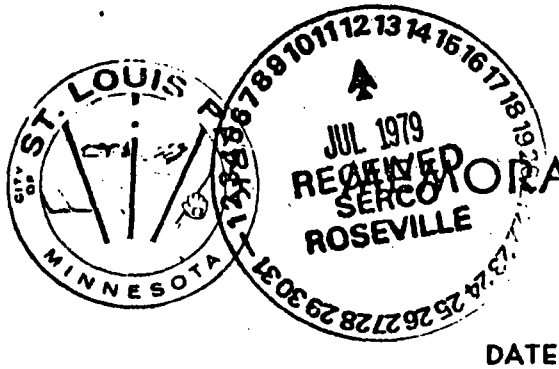
RAW

Concentration of Selected P.A.H. Compounds (ng/l)

COMPOUND	7/16 11:00 A.M 55037	7/18 11:00 A.M 55095	7/19 5:00 A.M 55102	7/20 5:00 A.M 55123
2-Methylnaphthalene	<67.	<67.	<67.	
Acenaphthene	3200.	2400.	2800.	<2.2
Biphenyl	* *	* *	* *	450.
Anthracene				60.
Phenanthrene	1700.	1800.	1900.	1450.
Pyrene	* * *	* * *	* * *	* * *
1,2,3,7-tetrahydro-pyrene				
Fluorene				
Fluoranthene	510.	440.	380.	190.
1,12-Benzanthracene	<52.	<52.	<52.	---
Chrysene	<25.	<25.	<25.	---
Benzo (a) pyrene	<2.2	<2.2	<2.2	<2.2
3,4-Benzophenanthrene	<15.	<15.	<15.	<1.0
Benzo (e) pyrene				
Perylene	<1.0	<1.0	<1.0	<1.0
Benzo (g,h,i) perylene				
Benzo (k) fluoranthene				
Benzo (a) fluoranthene				
Benzo (b) fluoranthene				
2-phenylenepyrene	<1.0	<1.0	<1.0	<1.0
1,2-5,6-Dibenzan-thracene	<38.	<38.	<38.	<1.0

*New column and data system installed on 7/26/79. Acenaphthene and Biphenyl no longer co-elute.
**Acenaphthene and Biphenyl co-eluted. The peak was calculated as Acenaphthene.
***Phenanthrene and Pyrene co-eluted. The peak was calculated as Phenanthrene.
--- Compound not in standard.

INTER-OFFICE MEMORANDUM



Dick Koppy DATE July 10, 1979

OM Vern Tollefsrud

BJECT Pilot Study - Carbon Treatment of Well Water Deep Well #15

On July 11, 1979, a meeting was held at the St. Louis Park City Hall. In attendance were Dick Koppy, Director of Public Works; Vern Tollefsrud, Water Superintendent; Bill Scruggins, Minnesota Health Department; Larry Briemhurst, Serco Labs and Darrel Thingvold of Serco.

The testing should begin on Monday, July 16, 1979. There will be two separate runs at different settings for the addition of the powdered carbon.

The M.H.D. will take about 20 to 25 samples.

Serco Laboratories will take about 7-10 samples plus P.H. and S.S. and temperature samples.

Some of these samples will be split samples. There will be two runs conducted. The first run will be with a projected 5 P.P.M. of carbon added to 1,000 G.P.M. of water. Sampling will be as follows:

First Run -- 5 P.P.M.

1. Raw water at Pump Head
2. Raw water effluent after filters
3. Six hour intervals for 48 hours of water effluent
4. Six hour intervals for 48 hours of suspended solids - carbon
5. Six hour intervals for 48 hours of temperature and P.H.
6. Backwash sample - composite of several cells.
7. Record pressure differentials in filter banks

Second Run -- 25 P.P.M.

1. Raw water at Pump Head
2. Raw water effluent after filters
3. Six hour interval for 48 hours of water effluent
4. Six hour intervals for 48 hours of suspended solids - carbon
5. Six hour intervals for 48 hours of temperature and P.H.
6. Backwash sample - composite of several cells.
7. Record pressure differentials in filter banks.

All effluent to be run into storm sewer system.
Turn around time for reading of samples about one week
Sample size to be 4 litre

If a third run is necessary to fine tune the application of carbon slurry, the same sampling times would be used.

A post testing meeting will be held at City Hall to review the results of this testing and further testing, using new carbon concentrations, may be conducted

Carbon Costs - $0.42 per pound
Samples Serco - $100.00 per sample - approximate
Deep Well #15 - Pumping 1,000 G.P.M.

Coordination of sampling and runs will be by Vern Tollefsrud

jme

CARBON TREATMENT PROCEDURES

1. Initial test run to be at least six days.

2. Set up equipment series 44 W & T slurry feeder

   slurry pump) - slurry mixer - flush lines - etc.
   slurry tank)

3. Start with initial dosage of 5 P.P.M.

   Recommended by Dr. Russ Frazier S.B.H. - may need more or less P.A.C.

4. Take sample after sand filter is completely purged.

   First sample about two hours into run

5. Take samples on set times - State Board of Health

   Samples to be taken at well head and after filtering.
   Bill Scrugging S.B.H. to do samples and timing of samples to be taken

6. Decrease or increase dosage to reach desired results.

   P.A.C. added or decreased as needed to bring P.A.H. into acceptable limits.

Federal regs on PAH may be out in June or July

7. Record pressure differentials on sand filters.

   To be sure that filters are operating at peak efficiency and as to how fast P.A.C. builds up on the filters.

8. Backwash when differential reaches #4 - #6 or the maximum time of 10 days.

9. All affluent shall be pumped to waste until given permission by the Health Department to place plant back into operation.

   It may require us to have more than one test run of two days or more.

INTER-OFFICE MEMO

SERCO LABORATORIES

To: St. Louis Park File **Date:** July 5, 1979

From: LHB

Re: Activated Carbon Pilot Plant Project to Remove PNA Compounds

---

Daryle Thingvold and I met with Dick Koppy and Vern Tollesfrud of St. Louis Park, and Bill Scrutin of the Minnesota Department of Health at St. Louis Park concerning this project. We reviewed the proposed pilot plant project and the test procedures as recommended by SERCO. Some changes were made in the proposed test procedure in order to keep costs down and to get by with the amount of powdered activated carbon the City has on hand (975 pounds).

At this time it was decided to make two test runs at concentrations of 5 parts per million powdered activated carbon and 25 parts per million powdered activated carbon. Each test run would last for two days. This requires a total of 720 pounds of activated carbon.

The following is the revised test procedures:

1. Before the start of the test program, backwash the filters and collect a sample of the raw water and filtered water.

2. Begin the test run of 5 parts per million powdered activated carbon and continue for two days. Sample the raw water two times per day (the sample collected in item 1 above counts for one of these samples), and sample the filtered water every six hours. This is equivalent to four raw water samples and eight filtered samples per two day test run. Collect the samples for PNA analyses in specially prepared bottles. Also collect the samples with the same frequency and at the same locations in general plastic bottles to be analyzed for pH and temperature. Some of the raw water samples will also be analyzed for suspended solids. The Minnesota Department of Health will provide all the necessary sampling containers for the PNA analyses.

   SERCO will provide the necessary PNA bottles for the following split samples: 1 raw water sample per run; 1 filtered water sample each day, or two per run; 1 composite backwash sample for one of the runs only.

3. Vern Tollesfrud will add a sampling tap on the filter influent line downstream from the point where the carbon is injected. Samples will be collected in a plastic container at six hour intervals, to correspond with the above sampling. These samples will be analyzed for suspended solids, temperature and pH. An attempt will be made to correlate these suspended solids results with the carbon feed.

4. Records will be kept of head loss through the filters at frequent intervals during each test run. Other records will be maintained for the amount of carbon used to mix up the slurry, amount of water used, the feed rate, etc.. Additional records will be kept of temperature, pH, flow rates, etc..

5. After two days the 5 parts per million feed rate will be discontinued, the filters will be backwashed, and the feed pump readjusted to feed at a rate of 25 parts per million. The next test run will begin immediately with the same sampling frequencies, etc. as above.

6. The following total numbers of samples will be collected by Vern Tollesfrud and/or the State Health Department:

Minnesota Department of Health Samples

Raw water	-	8 samples
Filtered effluent	-	16 samples
Backwash	-	2 samples
Total	=	27 samples

SERCO Split Samples for PNA analyses:

Raw water	-	2 samples
Filtered effluent	-	4 samples
Backwash	-	1 sample
Total	=	7 samples

SERCO Samples for Suspended Solids Analyses

Raw water 2/day x 4 days =	8 samples
Filtered samples 4/day x 4 days =	16 samples
Prefiltered samples 4/day x 4 days =	16 samples
Total general samples	40

In summary SERCO will provide a total of 40 general bottles and 7 PNA bottles, and the Minnesota Department of Health will provide 27 PNA bottles.

7. General comments:

- Vern Tollesfrud of St. Louis Park will be the project coordinator. It is planned to begin the project next Wednesday, July 11. Daryle will keep in contact with Vern and Bill Scrutin to coordinate SERCO's efforts.

- The composite sample of the backwash will be collected by taking equal portions of the sample every minute or two during one of the backwash cycles from one of the cells, once during each test concentration run. The sample will be filtered and the carbon will be extracted for PNA analyses.

- After the results from the two test runs are available, SERCO, the City and the Minnesota Department of Health will again meet to discuss the results. At this time a decision will be made whether to conduct a third test run. A final report will be prepared after this meeting. No discision was made who would prepare the report, but Dick Koppy implied he would like to do so, possibly with the advise of SERCO and the Health Department.

ks

cc: Daryle

Based upon a meeting at our office, the following recommendations are made for the initial carbon treatment testing:

1. Three predetermined levels of carbon concentrations should be used. Each concentration can be run over 2 days for a 6 day continuous period.

2. Sample raw and filtered water every four hours for a total of 12 raw and filtered water samples. Analyze samples for PNA, temperature and pH.

3. SERCO can analyze one split raw water and 2 split filtered water samples (one/day) for each run. Total number of samples analyzed by SERCO would be 3 raw water and 6 filtered water.

4. Sample every four hours downstream of the carbon injection point. Measure suspended solids, temperature and pH. Record head loss in the filter at least every four hours for each carbon concentration.

5. Record pumping rates.

6. Backwash at #4 - #6 differential and at the end of each of the three, 2 day runs. Backwash prior to starting the initial run.

7. Collect a composite sample of the backwash for each of the runs. Filter the water and extract the carbon for PNA compounds.

8. Run a statistical analysis of the data to determine the effect of carbon concentrations on PNA removal.

9. Possibly, initiate further testings using new carbon concentrations, etc.